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Study of the Silica Refractories

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DISCUSSION OF THIS PAPER IS INVITED. It should preferably be presented in person at the New York meeting, February, 1917, when an abstract of the paper will be read. If this is impossible, then discussion in writing may be sent to the Editor, American Institute of Mining Engineers, 29 West 39th Street, New York, N. Y., for presentation by the Secretary or other representative of its author. Unless special arrangement is made, the discussion of this paper will close Apr. 1, 1917. Any discussion offered thereafter should preferably be in the form of a new paper.

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A Study of the Silica Refractories*

BY J. SPOTTS McDOWELL, B. S.†

(New York Meeting, February, 1917)

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* An investigation conducted by the author at the laboratories of the Massachusetts Institute of Technology from October, 1915, to February, 1916, and published with the consent of Professor H. O. Hofman, Acting Head of the Department of Mining and Metallurgy. The supplementary report represents work done later while associated with the Harbison-Walker Refractories Co. at Pittsburgh, Pa.

† Research Department, Harbison-Walker Refractories Co., Pittsburgh, Pa.

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Acknowledgments

To a very considerable degree I am indebted to the kindness of others for the material herein presented. The subject was suggested to me by Kenneth Seaver, Chief Engineer of the Harbison-Walker Refractories Co., Pittsburgh, Pa., and represents, in fact, the continuation of an investigation begun by him.*

The materials studied were furnished by the Harbison-Walker Refractories Co., and the test brick manufactured at the Hays Station plant of that company under the supervision of R. H. H. Pierce, Chief Chemist. The conditions under which the test brick were made were devised by members of the Harbison-Walker Refractories Co.'s engineering department.

Whatever value the microscopic determinations may possess is due entirely to the careful instruction and kindly counsel of Professor C. H. Warren of the Massachusetts Institute of Technology. I am particularly grateful to Victor Dolmage, who was extremely generous with his time in assisting with some of these determinations. The photomicrographs are the work of W. L. Whitehead. The crushing and cross-breaking tests were made with the assistance of I. H. Cowdrey of the Mechanical Engineering Department of the Institute of Technology. I wish to express my thanks to all those named, as well as to others who have rendered assistance in various ways.

Introduction

The magnitude of the thermal expansion of silica brick, and its inability to withstand rapid temperature changes, present problems of considerable importance in the manufacture of siliceous refractories.

In the study represented by the experimental data herein described, these problems are considered from the point of view afforded by the results of recent investigators: Fenner and his associates of the Geophysical Laboratory, Washington, D. C., who have established the stability relations of the silica minerals, and Endell and co-workers in Berlin, who have studied the changes brought about in the constitution of siliceous refractory materials upon the application of heat.

In the process of manufacture, the quartzite from which the brick is made changes in part into cristobalite and tridymite. The attempt is herein made to determine by microscopic methods the degree of transformation in various specimens of test bricks manufactured at slightly varying temperatures of burning, with varying coarseness of grain, and to determine the effect of repeated burning. The effect of variations

* Kenneth Seaver: Manufacture and Tests of Silica Brick for the Byproduct Coke Oven, *Trans.*, vol. 53, pp. 125–139 (1916).

in these conditions upon crushing strength and modulus of rupture has also been considered.

Coincidently, a study has been made, from the available literature, of the properties of the silica minerals and the silica refractories. From the knowledge so gained, combined with the experimental data regarding the constitution of the test bricks, deductions have been made as to the conditions of manufacture under which silica brick of decreased thermal expansion and increased power to withstand rapid temperature changes might conceivably be produced. It is hoped that these deductions may serve to indicate possible starting points for future investigations.

The first part of this report comprises data compiled from various publications. Although much of this has no bearing upon the specific problem in hand, it is believed to be of interest in any thorough study of the properties of siliceous refractory materials.

THE SILICA MINERALS

Stability Relations

The crystal modifications of silica important in this connection are quartz, tridymite and cristobalite, each of which possesses α and β phases. Any one of these three modifications may be converted into either of the others by appropriate heat treatment. The formation of tridymite seems always to require the presence of a flux or catalyzer, while cristobalite is formed in the absence of a catalyzer. The inversion temperatures[1] are as follows:

870°C ± 10 quartz $\rightleftarrows$ tridymite
1,470° ± 10 tridymite $\rightleftarrows$ cristobalite
575° α quartz $\rightharpoonup$ β quartz; 570° β quartz $\rightharpoonup$ α quartz
117° α tridymite $\rightharpoonup$ β_1 tridymite; 163° β_1 tridymite $\rightharpoonup$ β_2 tridymite; reversions on cooling not sharp.
274.6° — 219.7° α cristobalite $\rightharpoonup$ β cristobalite
240.5° — 198.1° β cristobalite $\rightharpoonup$ α cristobalite

At ordinary temperatures each mineral exists only in the α phase, changing into the β phase as the temperature is raised. The α to β transformations are all rapid, while considerable time is required for the transformation of one mineral into another.

In the presence of a flux, when any form of silica is heated a sufficient length of time below 870°, quartz is always formed;[2] between 870° and 1,470° tridymite and from 1,470° upward to the melting point of silica, cristobalite. *Heated without a flux*, the inversion from quartz to tridymite does not occur, but the transformation is direct to cristobalite,

[1] C. N. Fenner: Stability Relations of the Silica Minerals, *American Journal of Science*, ser. 4, vol. 36, p. 383 (1913).

[2] *Ibid.*, p. 358.

and the temperature of incipient formation of cristobalite is depressed to about 1,250°; under the same conditions, tridymite is converted into cristobalite, with the inversion temperature raised to about 1,570°. Fenner's experiments on quartz powder heated without a flux gave the following results:

108 hr. at 1,250°: a very small per cent. of cristobalite formed.
90 hr. at 1,360°: resultant product two-thirds cristobalite, one-third quartz.
1 hr. at 1,570°: transformation into cristobalite nearly complete.

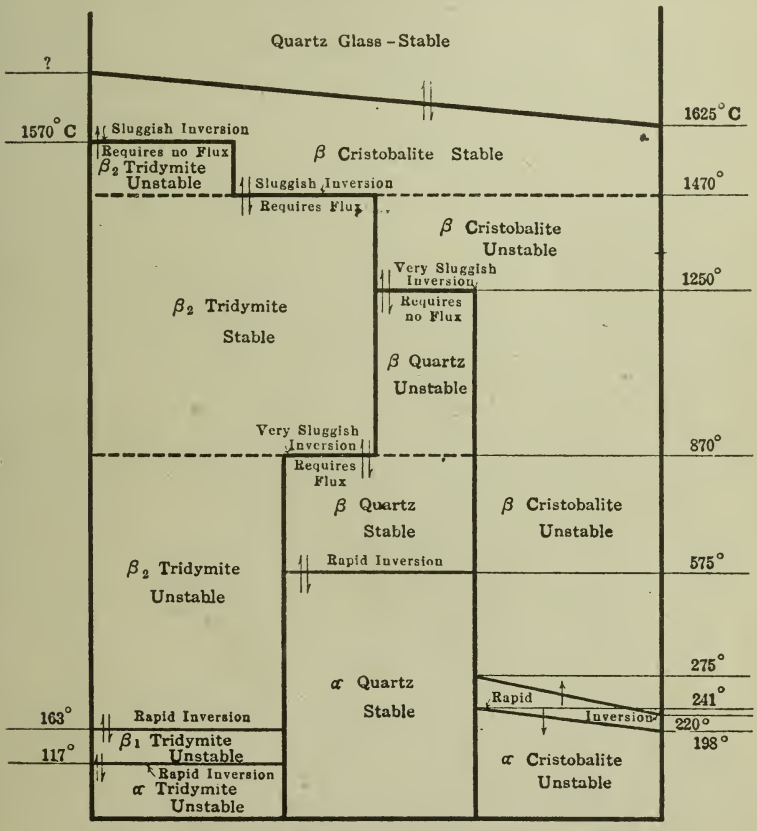


FIG. 1.—STABILITY RELATIONS OF THE SILICA MINERALS.

Quartz glass heated above 1,200° becomes gradually converted into cristobalite.[3] The percentages of cristobalite formed under various conditions of heating are shown in the accompanying table, taken from the work of Rieke and Endell.

[3] Rieke and Endell: Devitrification of Quartz Glass, *Silikat Zeitschrift*, vol. 1, No. 1, p. 8 (Jan., 1913).

TABLE 1.—*Cristobalite Formed by Heating Quartz Glass under Various Conditions (Rieke and Endell)*

Time of Heating, Hours	Temperature					
	1,200°	1,300°	1,400°	1,500°	1,600°	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	
½	—	—	5	34	60	
1	0	2	17	50	90	
2	0	5	25	100		
4	0	10	—			

Quartzite heated three to five times to 1,450° in a porcelain kiln is converted chiefly into cristobalite, with here and there scattered residual

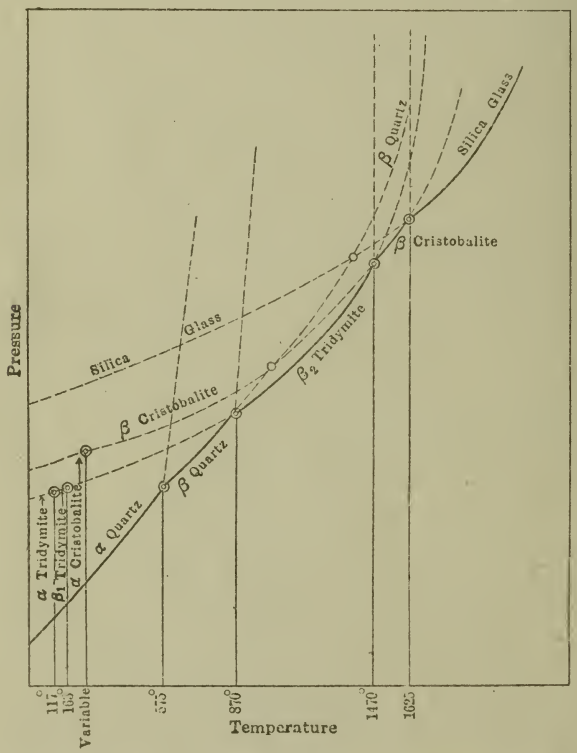


FIG. 2.—STABILITY RELATIONS OF THE SILICA MINERALS (AFTER FENNER).

quartz grains; after 10 burnings at 1,450° wedge-shaped twin crystals of tridymite begin to appear.[4] Silica brick, which consists principally of

[4] K. Endell: *Stahl und Eisen*, vol. 33, p. 1855 (Nov., 1913).

cristobalite when placed in use, gradually changes at high temperatures in the furnace to tridymite, as explained later on in this paper.

The stability fields and existence ranges of the various forms of silica are shown in Fig. 1; an equilibrium diagram (after Fenner) in Fig. 2.

TABLE 2.—*Optical Properties**

Composition	Crystal System	Crystal Habit	Elongation	Optical Orientation	nNa		Optical Character	Remarks
					α	γ		
α Quartz.....	Hexagonal Trapezohedral Tetartohedral	Pyramidal	Y	$c = \gamma$	1.544	1.553	+	Stable below 575°.
β Quartz.....	Hexagonal Trapezohedral Hemihedral	Pyramidal	Y	$c = \gamma$			+	Stable between 575° and 870°. Inverts on cooling to α quartz.
α Tridymite...	Orthorhombic	Thin pseudo-hexagonal plates			1.469	1.473	+	Exists below 117°. Usually finely intergrown aggregates. Optic axial angle large.
β Tridymite...	Hexagonal	Hexagonal plates		$c = \gamma$			+	Exists only above 117°. Inverts on cooling to α tridymite.
α Cristobalite.	Probably tetragonal	Equaint grains		$c = \alpha$	1.484	1.487	−	Exists below 275°.
β Cristobalite..	Isometric	Equaint grains						Exists only above 198°. Inverts on cooling to α cristobalite.

* C. N. Fenner: *American Journal of Science*, ser. 4, vol. 36, pp. 351–356 (1913).
N. L. Bowen: *American Journal of Science*, ser. 4, vol. 38, p. 245 (1914).
Rankin and Wright: *American Journal of Science*, ser. 4, vol. 39, pp. 4 and 74 (1915).

Tridymite crystals usually occur as crystalline aggregates of random orientation, as broad, thin hexagonal plates, which appear as needles or laths when turned on edge, and as wedge-shaped twin crystals. The hexagonal plates of usual thinness appear perfectly isotropic when lying on the base; the needle or lath-like sections have weak double refraction, parallel extinction and negative elongation.

Cristobalite does not develop a well-defined crystal form but usually occurs as minute branching skeleton crystals, with the branches often showing octahedral terminal caps. The higher temperature form of cristobalite is isometric; on cooling it becomes weakly birefringent. The birefringence in thin sections is barely discernible with the sensitive tint plate.

TABLE 3.—*Density*

	Specific Gravity	
Quartz	2.65	
Tridymite	2.270	Artificial tridymite (Fenner).
	2.28	Natural tridymite (Mallard).
	2.32	(Endell).
Cristobalite	2.333	Artificial cristobalite (Fenner).
	2.34	Natural cristobalite (Mallard).
	2.33	(Endell).
Cristobalite	.....	At 1,500° has same density as quartz glass; at 300°, slightly greater density than quartz glass (Rieke and Endell).
Quartz glass	2.21	(Dana, Endell).
	2.194	(Schwarz).

MELTING POINTS

"It appears that the fusing point of quartz is lower than 1,470°, but that at this temperature it passes into cristobalite almost as rapidly as it melts. The fusing point of tridymite should lie between those of quartz and cristobalite."[5] "It is clear that the indicated melting point of cristobalite must be higher than 1,625°, the value found by Fenner, higher than 1,685° even, the value found by Endell and Rieke.[6] . . . . . As Dr. Fenner has suggested, cristobalite may have a variable molecular constitution and a similarly variable melting point according to the conditions under which it was formed."[7] Kanolt[8] finds that pure silica flows distinctly at 1,750°, which is, therefore, the apparent melting point and higher than the true melting point. Quartz glass softens at 1,500° and melts between 1,700° and 1,800°.[9]

THERMAL EXPANSION

The results of LeChatelier's[10] experiments are shown in Fig. 3; those of Day, Sosman and Hostetter[11] in Fig. 4.

[5] C. N. Fenner: Stability Relations of the Silica Minerals, *American Journal of Science*, ser. 4, vol. 36, p. 383 (1913).

[6] Endell and Rieke: *Zeitschrift für Anorganische, Chemie.*, vol. 79, pp. 239–259 (1913).

[7] N. L. Bowen: Ternary System: Diopside-Forsterite-Silica, *American Journal of Science*, ser. 4, vol. 38, p. 218 (1914).

[8] C. W. Kanolt: Melting Point of Fire-Bricks, *U. S. Bureau of Standards, Technologic Paper* No. 10, p. 14 (1912).

[9] A. E. Marshall: Fused Silica Ware, Its Manufacture, Properties and Uses, *Metallurgical and Chemical Engineering*, vol. 10, pp. 248–249 (Apr., 1912).

[10] LeChatelier: La Silice, *Revue Universelle des Mines*, ser. 5, vol. 1, p. 90 (1913).

[11] Day, Sosman and Hostetter: Determination of Mineral and Rock Densities at High Temperatures, *American Journal of Science*, ser. 4, vol. 37, pp. 1–39 (1914).

LeChatelier gives the mean linear expansion of quartz glass as $0.0_6 7$; Kaye[12] gives the following figures: 0° to 30°C., $0.0_6 42$; 30° to 100°, $0.0_6 53$; 100° to 500°, $0.0_6 58$; 500° to 900°; $0.0_6 50$; 900° to 1000°; $0.0_6 80$. Quartz glass, partly devitrified by long-continued heating at a high tem-

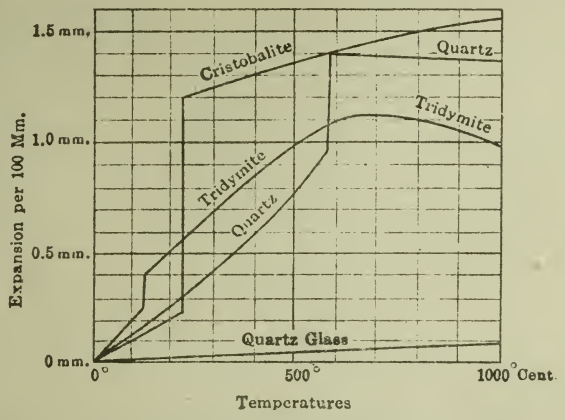


FIG. 3.—RESULTS OF LE CHATELIER'S EXPERIMENTS IN THERMAL EXPANSION.

perature, so that it contains much β cristobalite, if cooled rapidly to 300° remains completely clear and transparent, and but few cracks appear. When further cooled, however, to 230°, the sudden formation of numerous

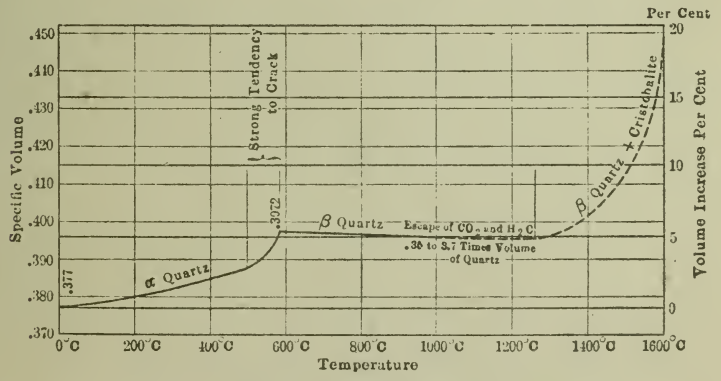


FIG. 4.—THERMAL EXPANSION OF QUARTZ.

fine cracks causes the glass to become white and opaque, coincident with the inversion of β to α cristobalite. The specific volume and coefficient of expansion of β cristobalite are, therefore, nearly the same as those of quartz glass; the presence of a few cracks at 300° indicate that the

[12] G. W. C. Kaye: Expansion and Thermal Hysteresis of Fused Silica, *Philosophical Magazine*, ser. 6, vol. 20, pp. 718–728 (Oct., 1910).

cristobalite probably has a slightly greater coefficient of expansion than the glass.[13]

Solubilities

Rammelsberg,[14] and Lange and Milberg[15] studied the solubility of quartz, opal and amorphous silica in alkaline solutions, but not that of tridymite or cristobalite. Cramer found the solubility in KOH of quartzite burned once in a porcelain kiln to be 41 per cent.; burned 10 times, 70 per cent. After the first burning, this material probably consisted of a mixture of cristobalite and quartz; after the tenth burning, of cristobalite and tridymite, with a small amount of quartz.[16] Schwarz[17] obtained the results shown in Table 4 on boiling powders with grains approximately 0.04 mm. in diameter.

Table 4

Reagent	Solubility of				Time, Hours
	Quartz, Per Cent.	Tridymite, Per Cent.	Cristobalite, Per Cent.	Amorphous Silica, Per Cent.	
5 per cent. Na_2CO_3 solution	2.11	2.77	....	....	½
5 per cent. HF solution...	30.17	76.30	74.3	96.6	½
1 per cent. HF solution...	5.20	20.30	25.8	52.9	1

Identification of Cristobalite and Tridymite

The method of identification most readily applied is by determination of the indices of refraction by the immersion method, described later on in this paper. Tridymite may also be recognized by its characteristic needles, laths and wedge-shaped twin crystals.

The methods of Rieke and Endell,[18] who have gone into this subject with great care, are given in the following paragraphs. Specific-gravity determinations were of little value in the case of quartz converted by heating into cristobalite or tridymite, for several reasons. The specific gravities of the two last-named are not greatly different: the material always contains some unaltered quartz; and the end products are so

[13] Rieke and Endell: Devitrification of Quartz Glass, *Silikat Zeitschrift*, vol. 1, No. 1, p. 6 (Jan., 1913).

[14] *Annalen der Physik und Chemie, Poggendorf*, vol. 112, pp. 177 (1861).

[15] *Zeitschrift für Angewandte Chemie*, 1897, pp. 393 and 425.

[16] K. Endell: *Stahl und Eisen*, vol. 33, 1770 *et seq.* (Oct., 1913).

[17] R. Schwarz: Chemical Relations of the Various Modifications of SiO_2, *Zeitschrift für Anorganische Chemie*, vol. 76, p. 424 (1912).

[18] Rieke and Endell: Volume Change of Some Ceramic Raw Materials on Burning, *Silikat Zeitschrift*, vol. 1, No. 4, pp. 67, 85 (Apr., 1913).

filled with microscopic cracks that the results of specific-gravity determinations are always low.

These fine cracks interfered with microscopic investigations. On account of total reflection at the borders, according to Rieke and Endell, the greater part of the end product appears isotropic, and the birefringence can be detected only upon very careful observation.

By the use of the thermal microscope, it was seen that cristobalite becomes isotropic at 225° to 230°. The effect was particularly evident in cristobalite derived from quartz glass or from quartz heated above 1,600°; it could not be used as a means of identification in the case of cristobalite formed below 1,450°, which, on account of the cracks

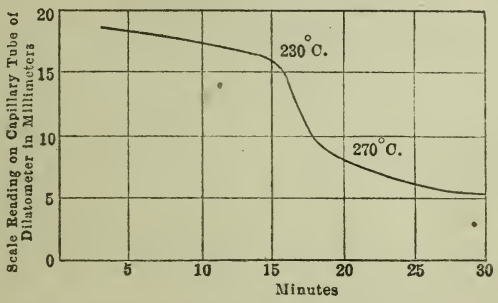


FIG. 5.—CURVE SHOWING EFFECT OF HEAT ON A SPECIMEN OF SILICA BRICK COMPOSED ESSENTIALLY OF CRISTOBALITE.

above mentioned, appears nearly isotropic. The change in volume of cristobalite at 230° is considerable. For that reason, quicksilver dilatometer observations offered a sure way of distinguishing cristobalite from tridymite. A specimen of silica brick, composed essentially of cristobalite, on heating[19] gave the curve shown in Fig. 5.

Rieke and Endell have designated the sudden clearing up shown by cristobalite heated carefully over the flame at about 230°, as the "cristobalite reaction." This effect can easily be observed with the naked eye in the case of cristobalite derived from quartz glass.

THE SILICA REFRACTORIES

RAW MATERIAL

American Deposits

The raw material used for the manufacture of high-grade American silica brick is a true quartzite; the deposits most extensively used are described by Seaver.[20] Named in the order of their importance, they

[19] *Stahl und Eisen*, vol. 33, p. 1856, Fig. 4 (Nov., 1913).

[20] K. Seaver: Manufacture and Tests of Silica Brick for the By-Product Coke Oven, *Trans.* vol. 53, pp. 125–139 (1916).

are the so-called Medina or Tuscarora sandstone of Huntingdon and Blair Counties, Pennsylvania, the Baraboo quartzite of the Devil's Lake

FIG. 6.—GANISTER FLOE IN THE GORGE OF THE JUNIATA RIVER, NEAR MOUNT UNION, HUNTINGDON CO., PENNSYLVANIA.

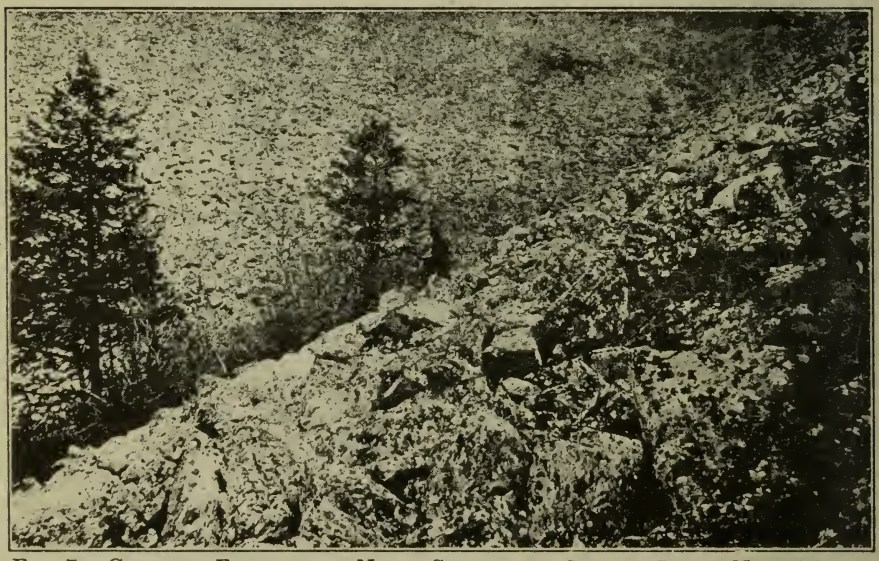


FIG. 7.—GANISTER FLOE ON THE NORTH SIDE OF THE JUNIATA RIVER, NEAR MOUNT UNION, HUNTINGDON CO., PENNSYLVANIA.

region, Wisconsin, and the deposits of eastern Alabama and of Colorado.

Where quarried for refractory purposes, the Medina is white, hard

and resistant. Its steeply pitching beds form the crests of many of the mountains of central Pennsylvania, and the steep slopes below the summit are covered by great bodies of talus, composed of blocks of the rock, varying in size from pieces weighing a few pounds to those weighing many tons. The talus slopes sometimes cover areas of a thousand acres or more where the beds have been cut through by streams. For the manufacture of silica brick, most of the rock used is taken from these bodies of loose rock, locally called "ganister floes," although rock from the solid measures is sometimes used.

Figs. 6 and 7 are pictures of ganister floes near Mt. Union, Huntington Co., Pa., where the Juniata River cuts through Jack's Mountain.

TABLE 5.—*Typical Analyses**

Constituent	Medina Quartzite from Pennsylvania	Baraboo Quartzite	Alabama Quartzite
SiO_2	97.80	97.15	97.70
Al_2O_3	0.90	1.00	0.96
Fe_2O_3	0.85	1.05	0.80
CaO	0.10	0.10	0.05
MgO	0.15	0.25	0.30
Alkalies	0.40	0.10	0.31

* K. Seaver: *Op cit.*, p. 127–128.

Properties Determining Usability

Not all pure quartzites are adapted to the manufacture of refractories. For refractory purposes three properties are to be considered: mechanical strength, melting point and behavior on burning. Experience shows that to produce a physically strong brick the stone should be hard, dense, and give splintery, angular fragments of heterogeneous size and shape on crushing; one that crushes down to rounded grains can not be used. The strength of the brick and its melting point are conditioned by the analysis of the rock. The silica content may vary from 96 to 98 per cent.; if less silica is present the fusion point is lowered. Attempts to use pure quartzite of 99 per cent. SiO_2 have not met with success. The presence of Al_2O_3 and Fe_2O_3 is necessary to form a bond; their combined percentage is usually about 1.75 per cent., and should not exceed 2.5 per cent. The amount of alkalies should be less than 0.5 per cent.

The melting interval of the stone depends upon its content of bases. For good quartzite it is usually considered to be cone 35 to 36; that is, 1,755 to 1,775°C.[21] Endell[21] obtained the following results, measuring temperatures by means of an iridium-iridium-ruthenium thermoelement

[21] K. Endell: *Stahl und Eisen*, vol. 33, p. 1857 (Nov., 1913).

in the calibration of which the melting points of gold, palladium and platinum were taken as 1,063°, 1,549° and 1,755°, the values determined by Day and Sosman:[22]

At 1,650°C., fine powder of good quartzite sintered together; pea-sized pieces showed no signs of fusion.

At 1,690°C., fine powder of good quartzite melted together into drops; the pea-sized pieces were superficially melted with visible rounding of the edges.

At 1,710°C., little pieces of pure Brazilian quartz 1 to 2 mm. in cross-section were melted round after 10 min. heating; a piece of ¼-in. cross-section had strongly rounded edges. Pea-sized pieces of good quartzite were strongly vitrified and showed fully rounded edges.

Expansion on Heating[23]

Chemical analysis alone does not afford an adequate index to the behavior of a quartzite on heating. Material suitable for refractory purposes does not weaken or crack perceptibly under the influence of high temperatures, and shows considerable volume increase after a first burning, retaining essentially the same volume later after repeated burning. Unsuitable quartzites, notably the pure, coarsely crystalline variety, become badly cracked or completely disintegrated on heating; they usually expand but little on a first burning and considerably thereafter.

TABLE 6

Material	Volume Increase after Burns				
	1	2	3	4	5
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Good quartzite (average of three samples)	9.4	10.7	11.0	11.3	11.5
Quartzite unsuitable for refractory purposes (average of three samples)	5.3	6.5	6.5	9.7	10.7
Melting point of all samples cone 35 to 36.					

In Cramer's[24] experiments, hard, fine-grained quartzites, which did not weaken in the heat treatment, gave the greatest expansion on the first burn. The volume increase of one specimen of good quartzite heated

[22] Day and Sosman: *Zeitschrift für Anorganische Chemie*, vol. 72, pp. 1–10 (Aug., 1911).

[23] F. T. Havard: *Refractories and Furnaces*, p. 36 McGraw-Hill Book Co., New York, 1912.

[24] *Stahl und Eisen*, vol. 21, p. 772 (July, 1901); *Tonindustrie Zeitung*, vol. 25, p. 864 (1901).

to cone 16 (1,460°±) was 17.1 per cent. after the first burn, 22 per cent. after the sixth. This enlargement was due to two factors: (1) an increase in porosity and (2) an expansion of the mineral itself, resulting from the gradual transformation of the quartz into cristobalite and tridymite. Only the latter effect is shown in the figures of Table 6, calculated from specific gravities of samples of quartzite heated repeatedly to 1,450° in a porcelain kiln.[25]

Texture

The insufficiency of analyses and melting-point determinations to indicate the value of a given quartzite for refractory purposes led Wernicke and Wildschrey[26] to seek an explanation in extensive microscopic studies of the texture of the rock. Their conclusions are as follows:

Typical quartzites, consisting principally of differently oriented intergrown quartz grains fairly uniform in size, metamorphosed quartzites, or those showing undulatory extinction under the microscope, can not be used. Good quartzites consist of quartz grains, mostly rounded, in a groundmass or cement of amorphous silica or crypto-crystalline quartz. No muscovite was found in such quartzites. Since expansion upon heating presents no difficulties in the case of quartzites containing a cement, only their melting point need be considered. They expand without cracking and acquire nearly their whole expansion on the first burn. In consequence of the fineness of division of the impurities in the cement and the slight sintering caused thereby, they are much stronger after burning than the typical quartzites in which the impurities are not so finely divided. The latter crack on burning and attain their complete expansion more slowly.

Grum-Grzimailo,[27] on the other hand, expresses the opinion that any quartzite with more than 94 to 95 per cent. SiO_2 may be used. He states that the purer the material and the larger the single quartz grains the longer must be the time of burning; the finer the quartz and its impurities the more rapidly will the required changes take place on heating.

Wernicke and Wildschrey's views are not supported by microscopic examinations of the most important American quartzites as shown later in this paper.

Silica Brick

The material herein discussed is termed "silica brick" in American practice, and is made from quartzite of 96 to 98 per cent. SiO_2 content to

[25] Calculated by the writer from data of K. Endell: *Stahl und Eisen*, vol. 33, p. 1774 (Oct., 1913).

[26] Wernicke and Wildschrey: Quartzite and Its Application in the Refractories Industry, *Tonindustrie-Zeitung*, vol. 34, pp. 688, 723 and 768 (1910).

Wernicke: Quartzite and Silica Brick, *Stahl und Eisen*, vol. 33, p. 1860 (Nov., 1913).

[27] *Stahl und Eisen*, vol. 31, p. 225 (1911).

which about 2 per cent. of lime is added to serve as a bond. It is not to be confused with the so-called "quartzite" brick, which contains much less silica, and in which fire clay is the bonding material.

Manufacture

The processes of manufacture are discussed by Seaver,[28] Hofman,[28] and Havard.[28] After grinding in a wet pan, where the lime is added, the charge is molded into brick, dried, and burned in a down-draft kiln.

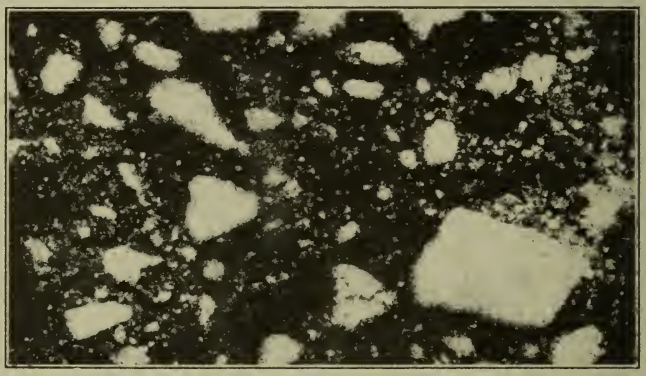


FIG. 8.—PHOTOMICROGRAPH OF POLISHED SURFACE OF 9-IN. SILICA BRICK. × 2

The fineness of grinding is determined by the character of brick to be made; the grind used for special shapes has a larger proportion of fines

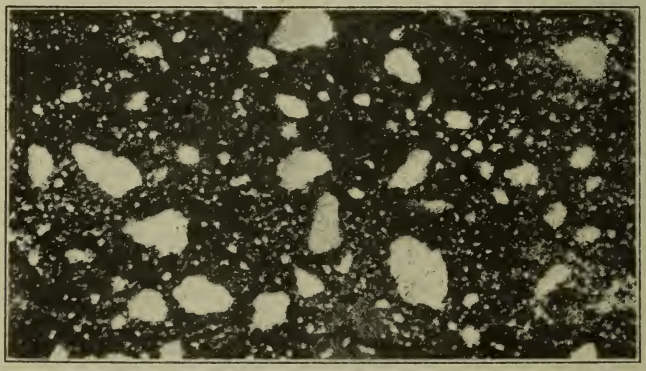


FIG. 9.—PHOTOMICROGRAPH OF POLISHED SURFACE OF SILICA SHAPE. × 2.

than that used for standard 9-in. brick. In either case, the ground material consists of a mixture of grains varying in size from that of a pea to the finest powder.[29] All silica brick are made in molds smaller than

[28] See Bibliography.

[29] See screen analyses, Table 13.

the proposed size of the finished brick; 3/8 to 7/16 in. per foot is allowed for expansion in burning.

The brick must be burned to such a temperature that as much as possible of the permanent expansion shall be acquired in the kiln, and not later in the furnace. The burning temperature is usually given as cone 15 to 16, or 16 to 17, and the time of burning as 20 days, including the time required for cooling. Lange[30] states that cone 15 should be melted down, cone 16 beginning to melt.

Properties of Silica Brick

Analysis.—A typical analysis[31] of brick made from Pennsylvania rock is as follows:

	Per Cent.
SiO_2	96.25
Al_2O_3	0.88
Fe_2O_3	0.79
CaO	1.80
MgO	0.14
Alkalies	0.39
	100.25

Brick with less than 94 per cent. SiO_2 or more than 3 per cent. CaO do not have the necessary refractory qualities; if less than 1.5 per cent. CaO is present, the brick are weak.

Density.—Principally on account of the progressive transformation of quartz into other modifications of silica upon the application of heat, the density of a silica brick is dependent upon the time and temperature of burning. Within certain limits, long-continued heating or burning at higher temperatures causes the density to diminish. The specific gravity of a silica brick should presumably lie between 2.65 (the value for quartz) and 2.28 (the value for tridymite). Microscopic cracks permeating the material render accurate determinations difficult, and results obtained in the ordinary way by the use of large pieces are quite sure to be inaccurate, even when the sample has been previously boiled.

For more accurate results, the finely powdered substance, boiled to remove adherent air bubbles, is usually tested in a pycnometer. Although somewhat laborious, this method with homogeneous materials gives results accurate in the second decimal place. As burned quartz is not homogeneous (see Figs. 16 and 17), and as the very fine cracks and pores are not always filled by the pycnometer liquid, the accuracy of the determinations in the case of silica brick is considered to be $\pm$ 0.02.[32]

[30] *Stahl und Eisen*, vol. 32, p. 1729 (Oct., 1912).

[31] K. Seaver: *Op. cit.*, p. 134.

[32] K. Endell: *Stahl und Eisen*, vol. 33, p. 1773 (Oct., 1913).

In this way Goerens and Gilles[33] determined the specific gravity of certain samples as 2.44. The figures given for the volume density of silica brick range from 1.50 to 1.88, averaging about 1.67 for a well-burned 9-in. brick. The values quoted by different writers for the "true specific gravity" of this material are quite inconsistent, varying from 2.05[34] to 2.75.[35]

Inconsistencies inexplicable by variations in the materials studied may perhaps be explained on the following grounds:

Let W = weight of specimen in grams.
V = entire volume of body in cubic centimeters.
V_s = volume of rock substance.
V_p = volume of pores.
V_o = volume of open pores, which absorb water.
V_c = volume of closed pores and cracks, which absorb no water.
$V_p = V_o + V_c$.
$V = V_s + V_p = V_s + V_o + V_c$.

It is evident that three possible values may be obtained for the specific gravity.

1. G_b. Bulk specific gravity $= \frac{W}{V} = \frac{W}{V_s + V_o + V_c}$.

2. G. True specific gravity $= \frac{W}{V_s}$. As already pointed out, for silica brick this value should probably lie somewhere between 2.28 and 2.65, depending on the conditions of burning.

3. G'. Apparent specific gravity, determined from the weights of dry specimen and water displaced after saturation. Apparent specific gravity $= \frac{W}{V_s + V_c}$. Its value lies between that of G and G_b and is dependent on the relation of closed to open pore space. When $V_c = O$, $G' = G$; when $V_o = O$, $G' = G_b$.

In many cases, the values quoted for the "true specific gravity" of silica brick are obviously too low. It is not improbable that in such cases the value designated above as G' has been considered to be the true density and so reported.

Porosity.—The porosity of silica brick varies within wide limits, dependent upon the methods of manufacture. The importance of this property lies in its relation to thermal conductivity and permeability

[33] Goerens and Gilles, *Ferrum*, vol. 12, p. 17 (Nov., 1914).

[34] F. T. Havard: *Op. cit.*, p. 37.

[35] Wologdine and Queneau: Conductivity, Porosity, and Gas Permeability of Refractory Materials, *Electrochemical and Metallurgical Industry*, vol. 7, p. 433 (Oct., 1909).

to gases.[36] Under ordinary conditions pore space acts as a heat insulator.[37] Following the notation of the preceding paragraph,

Total pore space in per cent. of volume of body $= P_t = \frac{100\,V_p}{V} = 100\,\frac{(1 - G_a)}{G}$.

Open pore space in per cent. $= P_o = \frac{100\,V_o}{V} = 100\,\frac{(1 - G_b)}{G'}$.

P_o is determined by absorption tests; P_t, by ascertaining the true specific gravity. Many writers upon silica brick do not make this distinction, but assume that $P_o = P_t$. The figures commonly given for porosity range from 18 to 43 per cent. of volume of the brick.

Strength.[38]—The figures in Table 7 are taken from Havard.

TABLE 7.—*Figures Taken from Havard**

Silica Brick Made in	Crushing Strength, Pounds per Square Inch		
	Side	Edge	End
Illinois	2,345	1,546	2,119
Pennsylvania	2,896	1,044	1,551
Missouri	2,370	1,792	1,803

Seaver[39] studied the effect of reburning on crushing strength and modulus of rupture with silica bricks made in Pennsylvania burned repeatedly to 1,540°±. His results (Table 8) show that with brick burned three times, the strength of the brick is increased by each reburning.

TABLE 8

Number of Burns	Average Modulus of Rupture, Pounds per Square Inch. Brick Stood on Edge, Supports 6 In. apart	Average Crushing Strength, Pounds per Square Inch. Brick Tested Flatwise
1	624	4,313
2	809	4,396
3	1,001	4,500

At high temperatures, the strength of a brick is much less than at

* F. T. Havard: *Op. cit.*, p. 37.

[36] Wologdine and Queneau: *Op. cit.*
Goerens and Gilles: *Op. cit.*

[37] Dougill, Hodsman and Cobb: Thermal Conductivity of Refractory Materials, *Journal of the Society of Chemical Industry*, vol. 34, pp. 465–471 (May, 1915).

[38] See also under Cross-breaking and Crushing Tests.

[39] K. Seaver: *Op. cit.* p. 138.

ordinary temperatures.[40] Subjected to a load of 50 lb. per square inch and gradually heated, chrome bricks fail suddenly at 1,450°, magnesia bricks at 1,550°. Silica bricks under the same conditions are unaffected at 1,450°.

Melting Point.—The melting interval of silica brick is generally considered to lie between cones 34 and 36 (1,740 to 1,775° C.)[41]; Havard[42] states that silica brick softens between 1,750 and 1,800°. Kanolt[43] measured the temperature at which pieces of hazelnut size visibly changed their form. For three different pieces the temperatures were 1,700°, 1,705°, 1,700°. Endell[44] tested brick consisting essentially of cristobalite, as well as brick consisting essentially of tridymite. Pea-sized pieces of both became completely melted at 1,700° ± 10.

The apparent discrepancy between the melting temperature of silica brick as determined in the laboratory, and the temperatures to which they are sometimes subjected in practice, is explained as follows:[45] Only the immediate surface of the brick reaches the furnace temperature. This is soon melted when 1,700° is exceeded, forming a viscous glaze, which protects the portion of the brick immediately adjacent. This portion does not reach its melting point on account of the temperature drop toward the exterior of the furnace.

TABLE 9.—*Specific Heat of Silica Brick**

	Heyn		Steger	Norton†
$t°$	True Specific Heat at $t°$	Mean Specific Heat between 20° and $t°$	Mean Specific Heat between 0° and $t°$	Mean Specific Heat between 20° and $t°$
0° C.	0.200	0.200		
200° C.	0.237	0.219	0.205	
400° C.	0.270	0.237		
600° C.	0.282	0.250		0.225
800° C.	0.285	0.261		0.236
1,000° C.	0.298	0.262		
1,100° C.				0.255
1,200° C.	0.291	0.266		

* E. Heyn: Thermal Conductivity of Refractory Building Materials, *Mitteilungen aus dem Königlichen Materialprüfungsamt*, vol. 32, p. 180 (1914).

W. Steger: Specific Heats of Refractory Products, *Silikat Zeitschrift*, vol. 2, No. 11, p. 203 (Nov., 1914).

† C. L. Norton: Private communication to K. Seaver.

[40] G. H. Brown: Note on Load Tests Made on Magnesite, Chrome and Silica Brick, *Transactions of the American Ceramic Society*, vol. 14, pp. 391–393 (1912).

[41] K. Endell: *Stahl und Eisen*, vol. 33, p. 1856 (Nov., 1913).

[42] F. T. Havard: *Op. cit.*, p. 29.

[43] *U. S. Bureau of Standards, Technologic Paper* 10 (1912).

[44] K. Endell: *Op. cit.*, pp. 1857, 1858.

[45] K. Endell: *Op. cit.*, pp. 1857, 1858.

Thermal Conductivity.—The divergence in the results obtained by different observers in the determination of thermal conductivity has occasioned considerable discussion. As Dudley points out, these variations are due to variations in the materials tested, as well as to differences between, or inaccuracies of, the methods employed. For comparative purposes, the coefficients are given in Table 10 for some fire-clay brick also. The coefficient k represents the flow of heat in calories per second, per square-centimeter area, through 1-cm. thickness, for a temperature difference of 1° C. To convert these figures into B.t.u. per 24 hr. per square-foot area per inch thickness per 1° F. temperature difference, they should be multiplied by 69,700.

TABLE 10

Material	Conductivity: Temperature t_1	Conductivity: Temperature t_2	Conductivity: Mean k between t_1 and t_2	Reference
Silica brick, "Star" brand 95.9 per cent. SiO_2	0	100	0.0021	Calculated from results of Boyd Dudley, Jr.
	0	1,000	0.0031	
Clay brick, "Woodland" brand 52.9 per cent. SiO_2, 42.7 per cent. Al_2O_3	0	100	0.0016	*Transactions of the American Electrochemical Society*, vol. 27, p. 336 (1915).
	0	1,000	0.0025	
"Quartzite" brick, 73.9 per cent. SiO_2, 22.9 per cent. Al_2O_3.	0	100	0.0020	
	0	1,000	0.0027	
Silica brick, 96 per cent. SiO_2, specific gravity, 2.44	0	100	0.0028	Calculated from results of Goerens and Gilles, *Ferrum*, vol. 12, pp. 1, 17 (Oct., Nov., 1914).
	0	1,000	0.0031	
Claybrick, 53.9 SiO_2, 40.2 Al_2O_3	0	100	0.0022	
	0	1,000	0.0027	

Notwithstanding the lack of numerical correspondence in results, Dudley, as well as Goerens and Gilles, finds that the coefficients are greater at high temperatures than at low, and that the conductivity of silica brick is greater than that of clay brick. The latter conclusion is corroborated by Brown,[46] who compared the flow of heat through the following materials:

Silica brick.....95.5 per cent. SiO_2, 1.9 per cent. Al_2O_3.
Quartzite brick.....83.4 per cent. SiO_2, 11.8 per cent. Al_2O_3.
Clay brick.....63.2 per cent. SiO_2, 33.0 per cent. Al_2O_3.

Cylindrical specimens were heated at one end to 1,300° C. while the other end was exposed to the atmosphere at 31° C. Under these condi-

[46] G. H. Brown: Relative Thermal Conductivities of Silica and Clay Refractories, *Transactions of the American Ceramic Society*, vol. 16, pp. 382–385 (1914).

tions, after equilibrium had been attained, the conductivity of the silica brick was 9 per cent. greater than that of the clay brick, and 3 per cent. greater than that of the quartzite brick.

Wologdine[47] shows that the conductivity of both silica and clay refractories increases with higher temperatures of burning. His figures indicate that silica is a poorer conductor than clay, for material burned at, or lower than 1,300°. Marshall[48] quite properly holds that while Wologdine's data are probably correct for the materials investigated, his figures are not practically applicable in a comparison of American refractories, since commercial silica brick are burned at a higher temperature than 1,300°.

The data given by Boyd Dudley, Jr.,[49] are of particular interest, in that the materials studied are three well-known brands of American refractories. The mean conductivities between any two temperatures t_1 and t_2 can be calculated from his results expressed by the following straight-line formula based upon measurements taken up to about 1,000° C.:

"Woodland" brick.....$k = 0.0015 + 0.0_510\ (t_1 + t_2)$
"Quartzite" brick.....$k = 0.0019 + 0.0_508\ (t_1 + t_2)$
"Star" silica brick.....$k = 0.0020 + 0.0_511\ (t_1 + t_2)$

These formulæ should be used with caution above 1,000°, and the formula for silicia brick should probably not be applied above 1,050°, as Heyn[50] finds a sharp rise in the conductivity of this material between 1,050° and 1,100°.

Expansion.—The problem of the expansion of silica brick is thus stated by Seaver:[51] In the process of manufacture, a permanent expansion occurs under the influence of heat, swelling the brick from the mold or "green" brick size to the required or "burned" size. It is desired that the permanent expansion should, if possible, be completed in the burning, so as to prevent further swelling in subsequent use. "In every burned brick expansion will occur on heating—a true thermal expansion, identical in its general character with that incident to the heating of practically all bodies. Such thermal or temporary expansion will disappear upon cooling, and being the expression of the coefficient of expansion of the silica it is unavoidable. This thermal or temporary expansion occurring upon heating a burned brick is not to be confused with the permanent expansion produced in the body of the brick during its manufacture. It is argued that if a silica brick is not properly burned,

[47] *Electrochemical and Metallurgical Industry*, vol. 7, pp. 383 and 433 (Sept.–Oct., 1909).

[48] S. M. Marshall: Relative Thermal Conductivity of Silica and Clay Brick, *Metallurgical and Chemical Engineering*, vol. 12, p. 74 (Feb., 1914).

[49] See Table 10.

[50] *Mitteilungen aus dem Königlichen Materialprüfungsamt*, vol. 32, p. 181 (1914),

[51] K. Seaver: *Op. cit.*, p. 133.

then, upon reheating in actual coke-oven practice, there will be not only a normal thermal expansion, but an additional expansion of indeterminate amount which will be permanent. It is essential in the construction of the coke ovens that the expansion, whatever its nature, be as small as possible, and that, whatever it is to be, it be known and adequately cared for."

Since the final increments of permanent expansion are acquired but slowly under the influence of high temperatures it is found that reburned brick show a slight increase in volume after each of several heat treatments. It is generally held that the manufacturer can do nothing to diminish the temporary expansion.[52]

The linear expansion in the burning from "green brick" size to size of finished product is $\frac{3}{8}$ to $\frac{7}{16}$ in. per foot, that is, 3.1 to 3.6 per cent. It may be here noted that the change of volume in the complete transformation of quartz to cristobalite corresponds to a linear expansion of 4.35 per cent.; quartz to tridymite, 5.3 per cent.

With a number of brick burned repeatedly to cone 16 to 17, Cramer[53] found an average permanent linear expansion of 3.2 per cent. on the first burn, an increment of 0.6 per cent. on second burn and 0.2 per cent. on the third. In a test mentioned by Havard,[54] a Pennsylvania silica brick reheated to 1,400° C. had a total linear expansion of 1.7 per cent., of which 0.4 per cent. was permanent, 1.3 per cent. temporary. Stockman and Foote,[55] at the Massachusetts Institute of Technology, studied the temporary thermal expansion of two brands of silica brick with the results shown in Table 11.

TABLE 11

Brand	Average Total Linear Expansion at			
	300°,	600°,	900°,	1,200°,
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
I. Reese & Sons	0.9	1.5	1.9	
Star	...	0.7	0.9	1.1

Practical experience shows that the thermal expansion of well-burned brick amounts to $\frac{3}{16}$ to $\frac{1}{4}$ in. per foot (1.6 to 2.1 per cent.) of which the greater part occurs below 600° C.

"In laying silica brick it is important to leave room for expansion;

[52] Experimental results hereinafter given indicate that this view is subject to certain qualifications.

[53] *Tonindustrie Zeitung*, vol. 25, p. 876 (1901); *Stahl und Eisen*, vol. 21, p. 772 (July, 1901).

[54] F. T. Havard: *Op. cit.*, p. 37.

[55] Massachusetts Institute of Technology Mining Department Thesis No. 226, 1902.

thus in a reverberatory furnace for melting sulphide-copper ores, having 12-in. silica brick, ¼ in. is allowed to 1 ft. in the roof, 5/16 in. to 2 ft. in the bottom; the expansion space is occupied by cardboard when the brick is being laid."[56]

Effect of Rapid Temperature Change.—Closely connected with the expansion on heating is the inability of silica brick to withstand rapid changes of temperature. For this reason, both while being manufactured and later when put into use, heating and cooling must take place evenly and slowly. "By quick burning" (in the kilns) "brick may be easily expanded by double or treble the normal amount; but sound brick do not result. This excessive expansion is due to minute fissures opened up in the body of the brick . . . In the early stages of firing the heat must be raised very slowly."[57] If cooled too rapidly, silica brick become friable, crack and spall. An underburned brick is less likely to spall than a well-burned one, but is weak, and has an excessive expansion, largely of a permanent character, when heated in the furnace.

Constitution

The constitution of silica brick and its relation to the phenomena of expansion have engaged the attention of many investigators. K. Endell[58] gives a historical sketch of the most important work done in this connection. In 1890, E. Mallard[59] discovered tridymite crystals in brick which had been in use a year and a half; this observation was confirmed in 1911 by Grum-Grzimailo.[60] The latter found that silica brick, when first made, consisted of grains of unaltered quartz surrounded by an apparently amorphous groundmass, which was held to be glass, together in some cases with varying amounts of tridymite; upon several months' use in the open-hearth furnace, the brick gradually and completely went over into a mass of interlocking twin crystals of tridymite. To avoid continual expansion in the furnace it was proposed to burn the brick in such a way as to effect the complete transformation to tridymite in the kilns.

Holmquist[61] discovered cristobalite in brick which had melted and hung down from the roof of the furnace as stalactites.

Endell[62] shows that the texture and constitution of silica brick are quite different before use, after 10 charges in the open-hearth furnace, after 50 charges, and again after having been melted. After the first burn, about two-thirds of the material was found to be converted into an

[56] H. O. Hofman: *General Metallurgy*, p. 368, McGraw-Hill Book Co., New York, 1913.

[57] K. Seaver: *Op. cit.*, p. 139.

[58] *Stahl und Eisen*, vol. 32, p. 392 (Mar., 1912).

[59] *Bulletin, Société française de minéralogie*, vol. 13, p. 172 (1890).

[60] *Stahl und Eisen*, vol. 31, pp. 224–226 (Feb., 1911).

[61] *Tonindustrie Zeitung*, vol. 35, p. 1325 (1911).

[62] *Stahl und Eisen*, vol. 32, p. 392–397 (Mar., 1912).

apparently isotropic modification of silica, enclosing unaltered quartz grains of unequal size. This apparently isotropic form was at first believed to be glass, but later shown to be cristobalite.[63] Since the transformation of the original quartz was not complete in the initial burn, the conclusion was reached that only about two-thirds of the expansion takes place in the kilns, and the rest must occur in the furnace. In a brick which had been through 10 charges in the open-hearth furnace, the groundmass was found to be converted to a large degree into tridymite, while the larger quartz grains were transformed into the apparently isotropic condition (cristobalite). After about 50 charges, a good silica brick consisted almost wholly of interlaced tridymite crystals. A brick was examined in which a rind 2 or 3 cm. thick had been melted, while the rest of the piece showed no signs of fusion. Three zones could be distinguished. The one lying nearest the heat had been fully melted, and later, in cooling, crystallized out in part as cristobalite, which was surrounded by a yellow iron-containing glass. The birefringence and other crystal properties of cristobalite formed in this way were much more easily recognized than with cristobalite formed by the transformation of quartz. The colorless cristobalite grains were penetrated by pearly cracks and twinned in a very complicated manner. In the thermal microscope they became isotropic at 220°. Analysis of this fused zone showed a silica content of but 90 per cent.; vaporization of SiO_2 had presumably been favored by intermediate formation of silicon carbide. The central zone showed the typical constitution of a tridymite brick which still contains some cristobalite. As the outer zone was approached, grains of quartz appeared; the latter zone contained considerable unaltered quartz in a groundmass of cristobalite, and in constitution appeared like an unused brick. It had evidently been kept cool by the outside air.

Endell[64] studied also the changes in the constitution of quartzites after repeated burning in a porcelain kiln to 1,450° C. It was found that the finely divided impurities of the quartzite serve as inversion centers and hasten the transformations.

Rock heated three times was converted principally into cristobalite, with some unaltered quartz grains remaining. The latter went over into cristobalite on longer heating and caused a continual expansion. After 10 heatings, there were still a few unaltered quartz particles, and a small proportion of tridymite had appeared in the form of the characteristic wedge-shaped twin crystals. Quartzite burned four times in the porcelain kiln, and later heated 2 hr. to 1,600° was completely transformed into cristobalite.

Seaver and Klein[65] made quantitative determinations of the constitution of silica brick and quartzite burned repeatedly to 1,540° C.,

[63] K. Endell: *Stahl und Eisen*, vol. 33, p. 1856 (Nov., 1913).
[64] *Op. cit.*, p. 1856.
[65] K. Seaver: *Op. cit.*, p. 137.

that temperature being maintained each time for about 40 hr. Their results are shown in Table 12.

TABLE 12

	Silica Brick			Quartzite	
	Burn			Burn	
	1, Per Cent.	2, Per Cent.	3, Per Cent.	1, Per Cent.	2, Per Cent.
Per cent. cristobalite.....	77	83	84	49	69
Per cent. quartz and silicates.....	23	17	16	51	31

No tridymite was to be expected in these tests, since that mineral is not known to be formed under any conditions above 1,470° C.

The relation of the constitution of silica brick to the expansion on heating is indicated by the facts that the volume increase in the transformation of quartz to cristobalite is 13.6 per cent.; quartz to tridymite, 16.7 per cent.

MICROSCOPIC STUDY AND EXPERIMENTAL DATA

Microscopic Examination of Quartzites

The accompanying photomicrographs represent the texture of specimens of quartzite from the deposits most important in the American refractories industry. All of these quartzites consist essentially of individual quartz crystals of random orientation closely packed together and interlocking with sharp angles. No trace of a groundmass or binding material is to be seen, except in a few small and isolated areas, which do not appear in the figures. Small inclusions are visible in great number. The accessory minerals are extremely small in amount, and have not been studied.

Fig. 10 shows the Medina quartzite from Mount Union, Huntingdon Co., Pa. This consists essentially of a mass of interfering quartz crystals, each with a sand grain as a nucleus. The rounded or subangular outlines of the original sand grains are marked by a row of inclusions, and are perfectly distinct. The spaces between these are filled with secondary quartz which has attached itself to the original grains in crystalline continuity with them, so that the original quartz grain and the new quartz border have the same extinction positions and interference colors. The borders contain but few inclusions, and consequently are much clearer than the nuclei.

The Baraboo Quartzite from Ablemans, Wis., is shown in Fig. 11. The grain is much coarser than that of the other quartzites examined, and the line along which the interlocking individuals join is peculiarly jagged. Many of the grains show a marked undulatory extinction with crossed nicols; that is, the whole of the crystal is not extinguished at

the same time, but a wave-like shadow passes over it on rotating the stage.

FIG. 10.

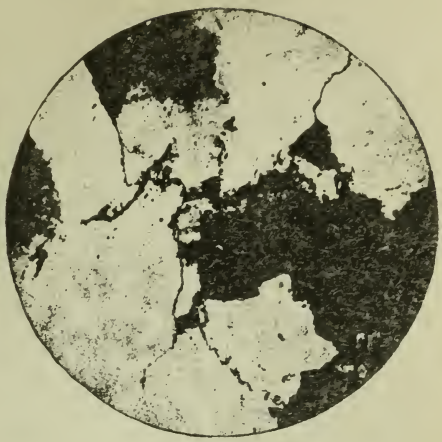


FIG. 11.

FIG. 10.—MEDINA QUARTZITE FROM MT. UNION, HUNTINGDON CO., PENNSYLVANIA (WHITEHEAD). THE FIELD SHOWS A MASS OF INTERFERING QUARTZ CRYSTALS EACH WITH A SAND GRAIN AS A NUCLEUS. THE OUTLINES OF THE ORIGINAL GRAINS ARE WELL SHOWN IN THE TWO CRYSTALS TO THE LEFT. CROSSED NICOLS. × 48.

FIG. 11.—BARABOO QUARTZITE FROM ABLEMANS, WISCONSIN (WHITEHEAD). UNDULATORY EXTINCTION IS SHOWN BY A GRAIN NEAR THE CENTER AND BY ONE NEAR THE TOP OF THE SECTION. CROSSED NICOLS. × 48.

FIG. 12.

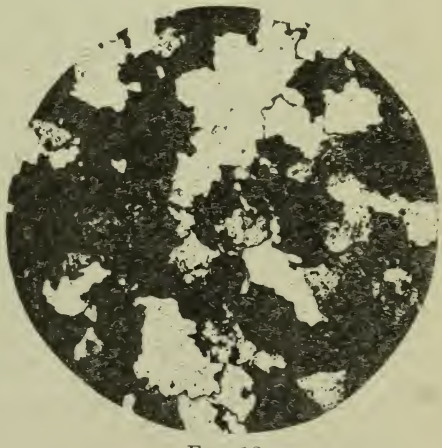


FIG. 13.

FIG. 12.—QUARTZITE FROM NORTHEASTERN ALABAMA, USED IN BRICK MAKING AT WYLAM, ALABAMA (WHITEHEAD). NOTE THE STRIATED APPEARANCE OF SOME OF THE GRAINS. CROSSED NICOLS. × 48.

FIG. 13.—QUARTZITE USED IN BRICK MAKING AT ANACONDA, MONT. (WHITEHEAD). CROSSED NICOLS. × 48.

The crystals of the Alabama quartzite contain numerous inclusions, arranged in nearly parallel lines, which with low magnifications appear under the microscope as fine striations. The Anaconda quartzite is extremely fine in grain.

The textural relations above described, and illustrated in the photomicrographs, do not sustain the conclusion[66] that only quartzites containing a fine-grained groundmass or cement can be used in the manufacture of high-grade silica refractories.

In Figs. 10, 11, 12, 13,[67] quartz grains in extinction positions are shown by black areas; quartz grains in other positions appear as lighter areas. The small black dots are mostly inclusions.

Study of Silica Brick

Description of Test Brick

The conditions under which the test brick were made were devised by the engineering department of the Harbison-Walker Refractories Co., with the purpose of studying the effects of variations in grind and temperature of burning, and of repeated burning, upon the physical properties and constitution of silica brick. Accordingly, three lots of 9-in. brick (approximately 9 by 4½ by 2½ in. in size) were made up. These lots will be hereinafter referred to as "regular", "medium", and "fine grind" respectively. The regular-grind specimens were made by the usual method for 9-in. brick; those of medium grind were given the grind used for silica shapes, and pounded into loose-side molds, as silica shapes are treated. The material used for the fine grind was screened through an 8-mesh hand screen, returned to the pan and reground long enough to add and mix lime thoroughly; the brick were pounded into loose-side molds. Screen analyses are shown in Table 13.

Table 13.—*Screen Analyses of Test Brick*

Grind	Screen Numbers in Meshes per Linear Inch					
	Held on					Through
	8, Per Cent.	16, Per Cent.	20, Per Cent.	30, Per Cent.	40, Per Cent.	40, Per Cent.
Regular	15.3	13.4	4.6	5.4	4.8	56.5
Medium or shape	10.2	13.1	2.2	5.0	4.7	64.8
Fine	.....	14.3	4.1	6.5	4.1	71.0

A number of brick from each lot were burned under the ordinary conditions of manufacture at three positions in the kiln, designated for convenience as positions *A*, *B* and *C*. The brick were burned a number of times, being under fire approximately 10 days each time, and specimens were set aside for study after each burning.

[66] See under *Texture*.

[67] Figs. 10 to 12 inclusive are from specimens furnished by the Harbison-Walker Refractories Co. Fig. 13 is from specimen furnished by the Anaconda Copper Mining Co.

The temperatures attained on the various burns as measured by Orton-Seger cones are shown in Table 14.

TABLE 14.—*Temperature Measured by Orton-Seger Cones*

Number of Burn	Position		
	A	*B*	*C*
1	Cone 15	Cone 13	Cone 14
2	Cone 15	Cone 13	Cone 14
3	Cone 15	Cone 15	Cone 15
4	Cone 13	Cone 14	Cone 14
5	Cone 15	Cone 15	Cone 15
6	Cone 16	Cone 14	Cone 14

The fusion points of these cones are as follows:

Cone	Orton Scale, Degrees Centigrade	Seger Revised Scale,* Degrees Centigrade
13	1,390	1,380
14	1,410	1,410
15	1,430	1,435
16	1,450	1,460

* Advertisement in 1914 Calendar of *Tonindustrie Zeitung*

The fusion points in silica brick kilns probably average 70° lower than the figures given by the manufacturers,[68] but can not be told with exactness, since cones give very erratic results when used in such kilns.[69]

Physical Tests

Density and Porosity.—The bulk and apparent specific gravities and per cent. of open pore space of certain brick of the first burn are shown in Table 15. The term "apparent" specific gravity, is used in the sense defined earlier in this paper under the heading, "Properties of Silica Brick." The composition of the brick (hereinafter discussed) suggests that the value of the true specific gravity, of which no determinations were made, probably lies between 2.40 and 2.45. The per cent. of open pore space was calculated from the two specific gravities (see discussion under heading just mentioned).

From the figures given it appears that the greatest porosity is shown by the coarse (regular) grind, and the least by the medium (shape) grind.

[68] Hoffman: *Tonindustrie Zeitung*, vol. 35, pp. 1099–1100 (1911).
S. Geijsbeek: Melting Points of Pyrometric Cones under Various Conditions, *Transactions of the American Ceramic Society*, vol. 14, 849–871 (1912).

[69] K. Seaver: *Op. cit.*, p. 133.

The data are insufficient to justify any conclusions as to the effect upon porosity of variations in burning temperature.

TABLE 15.—*Tabulated Data: Density and Porosity*

First Burn							
Mark	Grind	Burned to Cone	Bulk Specific Gravity	Apparent Specific Gravity	Open Pore Space, Per Cent. of Volume	Open Pore Space, Mean Per Cent. of Volume	Open Pore Space, Mean Per Cent. of Volume
83–A–1	Regular	15	1.678	2.259	25.7	26.1	
279–A–1	Regular	15	1.655	2.254	26.5		
69–C–1	Regular	14	1.637	2.229	28.5	28.5	
228–B–1	Regular	13	1.671	2.251	25.7	25.7	26.8
585–A–1	Medium	15	1.770	2.247	21.3	21.8	
601–A–1	Medium	15	1.748	2.249	22.3		
679–C–1	Medium	14	1.723	2.232	22.7	22.7	
443–B–1	Medium	13	1.839	2.277	19.2	19.2	21.2
1,148–A–1	Fine	15	1.757	2.272	22.7	22.5	
1,149–A–1	Fine	15	1.765	2.272	22.3		
854–C–1	Fine	14	1.725	2.242	23.2	23.2	
997–B–1	Fine	13	1.774	2.264	21.7	21.7	22.5

Cross-Breaking and Crushing Tests

In the transverse tests the brick were laid edgewise with a span of 6 in. and the load applied at midspan. Modulus of rupture was calculated from the formula $R = \frac{3\,Wl}{2\,bd^2}$, in which l is the distance between supports in inches, b the breadth and d the depth of the specimen in inches, and W the load in pounds at which failure occurred. Compression tests were made on half bricks resulting from the transverse tests. The bricks were bedded flatwise on blotting paper in the testing machine to secure a uniform bearing surface. The crushing strength was obtained by dividing the crushing load in pounds by the mean area in square inches of the two surfaces of the brick.

Unfortunately, but three brick of each lot were available for these tests, a number insufficient to permit the determination of the mean strengths with the proper degree of accuracy. The proposed specifications[70] of the American Society for Testing Materials direct that at least five specimens shall be used in transverse and compression tests upon building brick, and the same requirement should undoubtedly apply to the testing of refractory brick.

The results obtained are shown in Tables 16, 17, and 18.

[70] *Proceedings of the American Society for Testing Materials*, vol. 13, p. 284 (1913).

TABLE 16.—*Sample Log and Data Sheet*
Cross-Breaking and Crushing Tests

Cross-Breaking Span, 6 In.						Crushing: Half Brick Crushed Flat					
Mark	Breadth of Brick, In.	Depth of Brick, In.	*W*	*R*	Mean *R*	Crushing Area, Square Inches			*W*	*C*	Mean *C*
						1	2	Mean			
83–A–1	2.48	4.45	4,970	910		19.80	17.58	18.69	51,900	2,780	
279–A–1	2.46	4.45	4,820	890	920	19.54	17.97	18.75	45,600	2,430	2,680
289–A–1	2.52	4.43	5,260	960		16.46	16.16	16.31	46,100	2,830	
585–A–1	2.66	4.73	7,010	1,060		21.75	21.33	21.54	101,000	4,690	
601–A–1	2.66	4.71	7,450	1,140	1,050	21.18	20.24	20.71	120,500	5,820	4,780
715–A–1	2.68	4.71	6,320	960		20.26	20.00	20.13	77,000	3,830	

W = load causing failure in pounds.
R = modulus of rupture, pounds per square inch.
C = compressive strength, pounds per square inch.

TABLE 17.—*Collected Data*
Cross-Breaking and Crushing Tests

First burn — Brick Tested Edgewise 6-in. Span

Mark	Grind	Burned to Cone	Cross-Breaking Load in Pounds	Modulus of Rupture			Crushing Strength, Brick Laid Flat		
				Modulus, Pounds per Square Inch	Mean	Average Deviation, Per Cent. of Mean	Crushing Strength, Pounds per Square Inch	Mean	Average Deviation, Per Cent. of Mean
83–A–1	Regular	15	4,970	910			2,780		
279–A–1	Regular	15	4,820	890	920	3	2,430	2,680	6.4
289–A–1	Regular	15	5,260	960			2,830		
135–B–1	Regular	13	3,240	600			2,680		
228–B–1	Regular	13	3,630	670	690	10	2,390	2,730	8.5
327–B–1	Regular	13	4,280	800			3,120		
69–C–1	Regular	14	4,010	730			2,210		
102–C–1	Regular	14	4,140	750	770	5	2,050	2,100	3.3
156–C–1	Regular	14	4,520	820			2,040		
585–A–1	Medium	15	7,010	1,060			4,690		
601–A–1	Medium	15	7,450	1,140	1,050	6	5,820	4,780	14.0
715–A–1	Medium	15	6,320	960			3,830		
443–B–1	Medium	13	4,990	800			5,430		
445–B–1	Medium	13	4,310	690	910	24	3,890	4,680	11.0
476–B–1	Medium	13	7,760	1,230			4,710		
679–C–1	Medium	14	5,430	830			4,340		
680–C–1	Medium	14	5,610	860	920	11	4,930	4,820	6.7
744–C–1	Medium	14	7,050	1,070			5,180		
1,148–A–1	Fine	15	5,790	930			7,270		
1,149–A–1	Fine	15	9,450	1,500	1,170	19	4,360	5,880	17.0
1,151–A–1	Fine	15	6,770	1,070			6,010		
997–B–1	Fine	13	5,360	860			6,300		
998–B–1	Fine	13	6,180	990	910	6	5,280	5,840	6.3
999–B–1	Fine	13	5,560	890			5,940		
854–C–1	Fine	14	3,270	520			5,180		
934–C–1	Fine	14	2,840	450	790	52	5,040	5,250	3.4
1,052–C–1	Fine	14	9,010	1,410			5,520		

TABLE 17.—*Collected Data.—Continued*

Second burn

Mark	Grind	Burned to Cone	Cross-Breaking Load in Pounds	Modulus of Rupture			Crushing Strength, Brick Laid Flat		
				Modulus, Pounds per Square Inch	Mean	Average Deviation, Per Cent. of Mean	Crushing Strength, Pounds per Square Inch	Mean	Average Deviation, Per Cent. of Mean
275–A–2	Regular	15–15	5,820	1,080			2,230		
288–A–2	Regular	15–15	5,160	980			2,750		
290–A–2	Regular	15–15	3,480	620	910	15	2,990	2,680	8.6
293–A–2	Regular	15–15	5,140	940			2,760		
138–B–2	Regular	13–13	5,620	1,040			2,770		
218–B–2	Regular	13–13	4,730	860	1,010	10	2,540	2,760	5.4
330–B–2	Regular	13–13	6,070	1,120			2,970		
10–C–2	Regular	14–14	5,420	980			2,690		
14–C–2	Regular	14–14	4,730	880	920	3	2,490	2,680	4.6
110–C–2	Regular	14–14	4,950	920			2,850		
567–A–2	Medium	15–15	10,440	1,600			5,780		
599–A–2	Medium	15–15	10,730	1,630	1,680	5	7,010	6,030	11.0
628–A–2	Medium	15–15	11,800	1,810			5,290		
467–B–2	Medium	13–13	9,350	1,460			7,030		
472–B–2	Medium	13–13	10,390	1,630	1,310	24	5,200	6,270	11.0
579–B–2	Medium	13–13	5,360	840			6,590		
572–C–2	Medium	14–14	10,340	1,570			5,600		
658–C–2	Medium	14–14	10,620	1,620	1,680	7	5,130	5,570	5.2
699–C–2	Medium	14–14	12,150	1,860			5,970		
824–A–2	Fine	15–15	4,440	680			7,870		
848–A–2	Fine	15–15	4,830	770			7,850		
850–A–2	Fine	15–15	12,450	1,950	1,270	43	6,700	6,890	14.0
970–A–2	Fine	15–15	10,550	1,670			5,150		
961–B–2	Fine	13–13	5,420	860			5,610		
1,019–B–2	Fine	13–13	6,770	1,080	1,070	13	7,390	6,290	12.0
1,042–B–2	Fine	13–13	7,820	1,280			5,870		
805–C–2	Fine	14–14	13,650	2,160			8,560		
836–C–2	Fine	14–14	8,050	1,260	1,590	24	7,150	7,830	6.2
1,012–C–2	Fine	14–14	8,730	1,360			7,780		

TABLE 17.—*Collected Data.—Continued*

Third burn

Mark	Grind	Burned to Cone	Cross-Breaking Load in Pounds	Modulus of Rupture			Crushing Strength, Brick Laid Flat.		
				Modulus, Pounds per Square Inch	Mean	Average Deviation, Per Cent. of Mean	Crushing Strength, Pounds Per Square Inch	Mean	Average Deviation, Per Cent. of Mean
39-A-3	Regular	15-15-15	9,260	1,680			3,320		
53-A-3	Regular	15-15-15	4,060	740			3,000		
144-A-3	Regular	15-15-15	5,770	1,040	1,200	25	2,940	3,110	4.5
249-A-3	Regular	15-15-15	7,330	1,340			3,170		
174-B-3	Regular	13-13-15	7,190	1,310			3,000		
202-B-3	Regular	13-13-15	5,460	990	1,010	20	2,590	2,830	5.7
280-B-3	Regular	13-13-15	3,890	720			2,910		
66-C-3	Regular	14-14-15	6,290	1,170			2,650		
153-C-3	Regular	14-14-15	6,380	1,160	1,110	7	3,490	2,990	11.0
179-C-3	Regular	14-14-15	5,470	990			2,840		
427-A-3	Medium	15-15-15	4,910	770			6,570		
428-A-3	Medium	15-15-15	6,550	1,030			6,520		
439-A-3	Medium	15-15-15	7,840	1,230	1,210	25	6,890	6,550	2.8
501-A-3	Medium	15-15-15	11,680	1,820			6,220		
431-B-3	Medium	13-13-15	3,040	470			4,860		
568-B-3	Medium	13-13-15	10,520	1,610	1,320	43	6,220	6,380	17.0
622-B-3	Medium	13-13-15	12,280	1,880			8,070		
513-C-3	Medium	14-14-15	8,280	1,270			4,620		
672-C-3	Medium	14-14-15	12,790	1,950	1,580	16	4,520	4,830	7.0
674-C-3	Medium	14-14-15	9,770	1,510			5,360		
1,028-A-3	Fine	15-15-15	5,780	910			7,390		
1,046-A-3	Fine	15-15-15	6,340	1,000			6,920		
1,057-A-3	Fine	15-15-15	6,900	1,100	1,240	28	8,000	7,060	9.0
1,066-A-3	Fine	15-15-15	12,130	1,930			5,920		
846-B-3	Fine	13-13-15	13,900	2,180			8,680		
882-B-3	Fine	13-13-15	10,510	1,650	1,730	17	8,610	8,120	9.0
906-B-3	Fine	13-13-15	8,470	1,350			7,010		
880-C-3	Fine	14-14-15	6,800	1,070			7,070		
974-C-3	Fine	14-14-15	3,300	520	680	38	6,240	6,600	11.0
1,074-C-3	Fine	14-14-15	2,790	440			5,860		

TABLE 18.—*Summary*

Cross-Breaking and Crushing Tests

Burn	Burned to Cone	Modulus of Rupture, Brick Tested Edgewise, 6-in. Span						Crushing Strength, Brick Laid Flat					
		Regular Grind		Medium Grind		Fine Grind		Regular Grind		Medium Grind		Fine Grind	
		Mean	a.d.*	Mean	a.d.	Mean	a.d.	Mean	a.d.	Mean	a.d.	Mean	a.d.
1	15	920	3	1,050	6	1,170	19	2,680	6	4,780	14	5,880	17
	14	770	5	920	11	790	52	2,100	3	4,820	7	5,250	3
	13	690	10	910	24	910	6	2,730	8	4,680	11	5,840	6
2	15–15	910	15	1,680	5	1,270	43	2,680	9	6,030	11	6,890	14
	14–14	920	3	1,680	7	1,590	24	2,680	5	5,570	5	7,830	6
	13–13	1,010	10	1,310	24	1,070	13	2,760	5	6,270	11	6,290	12
3	15–15–15	1,200	25	1,210	25	1,240	28	3,110	5	6,550	3	7,060	9
	14–14–15	1,110	7	1,580	16	680	38	2,990	11	4,830	7	6,600	11
	13–13–15	1,010	20	1,320	43	1,730	17	2,830	6	6,380	17	8,120	9

* a.d. represents the average deviation in per cent. of mean.

Discussion of Cross-Breaking and Crushing Tests

Average Deviation and Index of Uniformity.—It is highly desirable not only that the brick should be mechanically strong, but also that different specimens of the same lot should be of uniform strength. For this reason, in Table 19, obviously the lot of brick of "regular grind, mark C–3," is a more satisfactory material, so far as cross-breaking is concerned, than the "fine grind, mark A–2," notwithstanding the fact that the mean modulus of rupture of the former is 1,110 lb. per square inch, of the latter, 1,270 lb. per square inch. Since, plainly, mean values in themselves are insufficient as a basis of comparison for the strength of such brick, the "average deviation" of the single observations from the mean (as developed in the principles of precision of measurements) has been calculated as a measure of uniformity of the different specimens, for each lot of brick tested.[71]

The deviation of single observations from the mean is due to two factors: (1) lack of precision of the measurements; and (2) lack of uniformity of different specimens of the material. In the series of tests herein described, variations due to the former factor amount to less than 2 per cent. and are practically negligible as compared with variations due to the latter. For this reason, the "average deviation" can be considered a true measure of uniformity. A very low "average deviation" is an indication of a high degree of

[71] Similarly, "average deviations" are given by the Mechanical Engineering Department of the Massachusetts Institute of Technology, in reporting results obtained in testing the strength of timber.

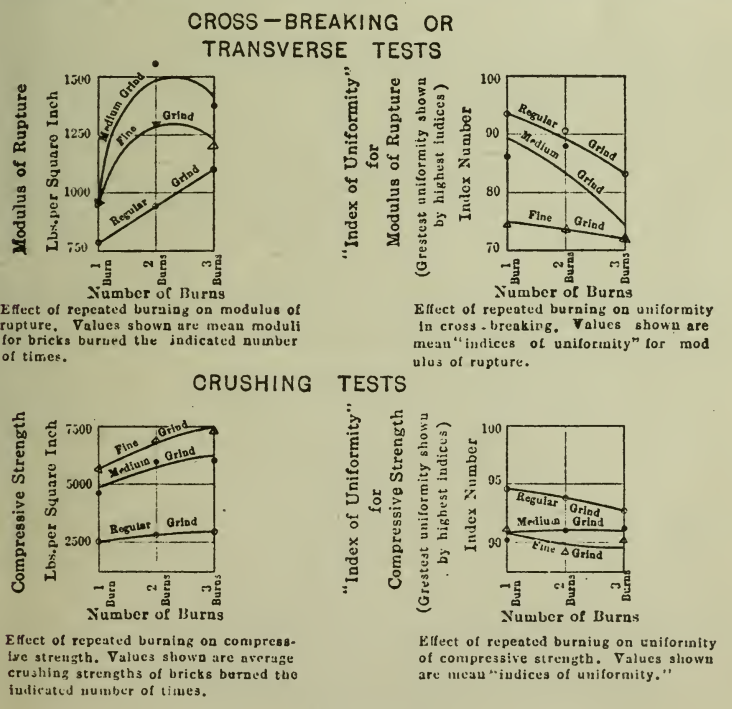
concordance in the values found for different specimens of the same material; a high "average deviation" indicates a lack of concordance.

The method of derivation of the "average deviation" for two specific cases is shown in Table 19.

TABLE 19

Grind	Mark	Modulus of Rupture	Mean Modulus	Deviation from Mean	Average Deviation or "a.d."	a.d. in Per Cent. of Mean
Fine.....	824–A–2	680		590		
Fine.....	848–A–2	770		500		
Fine.....	850–A–2	1,950	1,270	680	540	43.0
Fine.....	970–A–2	1,670		400		
Regular.....	66–C–3	1,170		60		
Regular.....	153–C–3	1,160	1,110	50	80	7.2
Regular.....	179–C–3	990		120		

In the graphical representation of the relative uniformity of different lots of specimens (Fig. 14) use has been made of an "index of uniformity,"



Effect of repeated burning on modulus of rupture. Values shown are mean moduli for bricks burned the indicated number of times.

Effect of repeated burning on uniformity in cross-breaking. Values shown are mean "indices of uniformity" for modulus of rupture.

Effect of repeated burning on compressive strength. Values shown are average crushing strengths of bricks burned the indicated number of times.

Effect of repeated burning on uniformity of compressive strength. Values shown are mean "indices of uniformity."

FIG. 14.

calculated from the average deviation (a.d.) thus: "Index of uniformity" = 100 − (a.d. in per cent. of mean). In the above table, the "index

of uniformity" for modulus of rupture is 57 on this scale for the first lot, 92.8 for the second.

Effective Ultimate Strength.—In comparing the strength of different lots of brick, their relative uniformity must be considered. As shown above, the mean ultimate strength is not a satisfactory basis for comparison. Since the "average deviation" is a measure of uniformity, it seems logical to consider the value obtained by subtracting the average deviation from the mean strength, as a more practical comparative standard. This value may be called the "effective ultimate strength;" and to this, rather than to the mean, the factor of safety should be

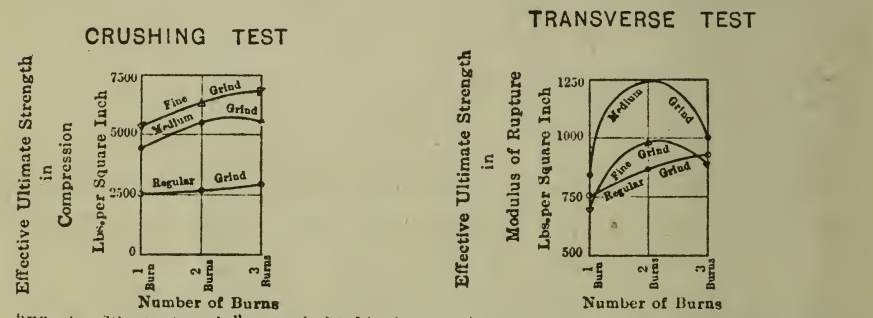


FIG. 15.

applied in calculating a safe working load for materials of which the load-carrying capacity is an important consideration. The effect of repeated burning upon the "effective ultimate strength," as so defined, is shown in Fig. 15.

Effect of Variation in Method of Manufacture.—The effects of the variables considered upon the strength of the test brick are represented graphically in Figs. 14 and 15.

The mean modulus of rupture of medium-grind brick is greatest; of regular-grind, least. The burning temperatures which produce the strongest brick in cross-breaking are as follows:

	First Burn	Second Burn
Regular-grind.....	Cone 15	Cone 15
Medium-grind.....	Cone 15	Cone 14 and 15
Fine-grind.....	Cone 15	Cone 14

Bricks of regular grind increase in cross-breaking strength after each burning; after the first burn, specimens burned at cone 15 are considerably stronger than those burned at cone 13 or 14, after the second burn this is no longer true. Modulus of rupture of medium- and fine-grind brick, in most cases, reaches a maximum on the second burn.

Crushing strength of regular-, medium- and fine-grind brick are to each other approximately as 41:84:100. There is an increase in strength after

each burn; a slight increase for regular grind, a considerable increase for medium and fine grinds.

Uniformity of strength of a given lot of brick diminishes with repeated burning; slightly in compression, considerably in cross-breaking. The most uniform brick are those of regular grind; fine-grind brick are fairly uniform in crushing, very non-uniform in cross-breaking strength.

The figures indicate that the medium-grind brick is the most satisfactory in point of strength, the regular-grind least so. While the compressive strength of the medium-grind is somewhat lower than that of the fine-grind, its cross-breaking strength is greater and it is a much more uniform material. In cross-breaking, the fine-grind shows little if any advantage over the regular-grind, for while its mean strength is high, it lacks uniformity; in compression, it is far stronger than the regular-grind.

There is, in all cases, an advantage in burning the brick a second time; for the medium and fine grinds, a third burn is usually harmful, decreasing the cross-breaking strength considerably, and increasing the compressive strength very slightly. Brick of regular grind increase in strength on each burn up to the third, slightly in compression, considerably in cross-breaking.

The alternate expansions and contractions incurred in repeated burning may tend to cause the formation of minute cracks in the brick and to have an unfavorable effect upon their strength. For that reason it is probable that stronger brick would be produced by maintaining the maximum temperature a greater length of time during a single burn than by resorting to repeated burning.

Microscopic Study of Constitution of Brick [72]

Method in General

Quantitative determinations of the proportions of quartz, tridymite and cristobalite in specimens of the various lots of brick were made by the Rosiwal micrometric method.[73] The measurements were all made upon powdered material, the minerals being identified through comparison of indices of refraction by immersion in liquids of definite indices. The Zeiss mechanical stage, and a lens system giving a magnification of about 390 diameters, were used.

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[72] The microscopic work was performed under the direction of Dr. C. H. Warren, without whose instruction and assistance the writer would not have ventured to undertake a study of this character.

[73] A. Johannsen: *Manual of Petrographic Methods*, p. 291. McGraw-Hill, New York, 1914. Lincoln and Rietz: Determination of the Relative Volumes of the Components of Rocks by Mensuration Methods, *Economic Geology*, vol. 8, pp. 120–139 (1913).

The indices of refraction of quartz are 1.544 to 1.533; cristobalite, 1.484 to 1.487; tridymite, 1.469 to 1.473;[74] calcium silicates, higher than those of quartz.[75] Separate portions of each powder were studied in two liquids whose indices of refraction were respectively 1.495 and 1.479. The higher-refracting quartz and compounds not identified were distinguished in the former liquid from the lower-refracting cristobalite and tridymite. A large part of the higher-refracting material had the refractive index and general properties of quartz.

The proportion of tridymite was determined by immersion of the powder in a liquid with refractive index of 1.479, which is between those of tridymite and cristobalite, but extremely close to them. On account of the latter fact, variations in room temperature caused some difficulty, since the index of the liquid was diminished 0.0005 to 0.0006 for each degree temperature rise. All material with an index lower than 1.479 was reported as tridymite, and the amount of cristobalite obtained by difference.

Sampling

A sample of about 300 grams from each lot of test specimens (100 grams from each brick of the lot) was crushed in a small Blake crusher and a disk grinder, to pieces approximately $\frac{1}{16}$ in. in diameter. After cutting down to ± 20 grams with a split shovel, the sample was reground until all passed through a 140-mesh sieve. For the microscopic study, that fraction was reserved (about 5 per cent. of the whole) which passed through a 240-mesh and was caught on a 260-mesh sieve. The size of these grains as determined microscopically varied from 0.05 to 0.06 mm. in diameter.

Identification

The principal optical property used in identifying the minerals was the index of refraction, by means of the Becke effect. The single grains were commonly made up of two, sometimes all three minerals, and the correct positions of the boundaries between tridymite and cristobalite could be told only by crossing the nicols and rotating the stage.

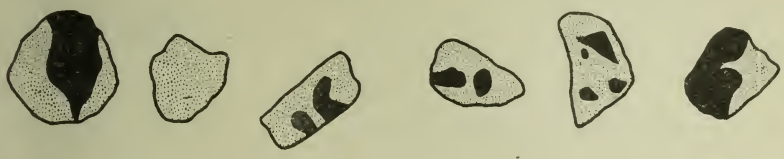
The more highly refracting material (chiefly quartz in the first burns) was easily recognized by its high relief in the liquid used, and by well-defined Becke lines. Tridymite was distinguished by its low index, low double refraction, and by the lath-shaped and wedge-shaped outlines of its crystals. Identification was confirmed by the facts that the lath-shaped sections had parallel extinction and negative elongation, and that

[74] See Table 2.

[75] Rankin and Wright: The Ternary System CaO–Al_2O_3–SiO_2, *American Journal of Science*, ser. 4, vol. 39, p. 74 (1915).

the index of refraction of the faster ray, as determined by the immersion method in sodium light, was 1.469.[76]

The low relief, the index of refraction, and the extremely low double refraction, which could usually be detected only with the aid of the sensitive tint plate, served to identify cristobalite. In the liquid with index 1.479, the Becke lines were faint, but their directions of motion could be



Quartz shown in black; cristobalite dotted

FIG. 16.—SKETCHES OF GRAINS OF POWDERED BRICK. FROM BRICK OF REGULAR GRIND, BURNED ONCE AT CONE 14. × 218 APPROXIMATELY.

detected upon close observation so long as the room temperature was kept at the proper point.

The lack of homogeneity of the grains led to the introduction of certain unavoidable quantitative errors. Quartz could, as a rule, be recognized even on the interior of the grains by the clearness of the Becke lines and the high relief; yet numerous small particles of residual quartz

Quartz shown in black; cristobalite dotted
tridymite cross-hatched

FIG. 17.—SKETCHES OF GRAINS OF POWDERED BRICK. FROM BRICK OF REGULAR GRIND, BURNED FOUR TIMES AT CONE 14–14–15–14. × 218 APPROXIMATELY.

were often so mingled with the cristobalite that an accurate quantitative separation of the two was impossible. The double refraction of tridymite was the only means of determining with exactness the boundaries between tridymite and cristobalite, but in sections of certain orientation could not be used. The presence of numerous microscopic cracks caused anomalous effects, and in many cases interfered with the Becke tests.

The texture of representative grains is shown in Figs. 16 and 17.

Precision

A coarser powder than that used was found to give less accurate results. The very fine portion, which went through the 260-mesh sieve,

[76] See C. N. Fenner: Stability Relations of the Silica Minerals, *American Journal of Science*, ser. 4, vol. 36, p. 351 (1913).

was exceedingly difficult to study. Since the latter fraction comprised about 85 per cent. of the original powder, it is evident that any great variation in its composition from that of the study sample will very seriously affect the results. For that reason, check determinations were made in a few cases of the amount of quartz in both the fraction which had passed through the 260-mesh sieve, and that which had been caught upon it. Results so obtained averaged 2 per cent. lower in the former case than in the latter, a difference which lies within the error of observation.

Errors introduced into the tridymite determinations by discarding the very fine material are probably serious, yet it was not found possible to obtain concordant results with this portion of the powder. As shown in Fig. 19, tridymite forms most rapidly in the fine-grained groundmass of the brick, and comparatively slowly in the larger grains. A much larger proportion of the groundmass than of the grains will undoubtedly crush down in the grinding so as to pass through a 260-mesh sieve. For that reason, the amount of tridymite in the fraction of the powder reserved for study, is probably considerably less than in the brick itself.

TABLE 20.—*Results of Microscopic Study—Tabulated Data Constitution of Test Brick*

Lot	Mark	Grind	Burned to Cone	Number of Burns	Constitution in Per Cent. by Volume		
					$n > 1.495$	$n < 1.479$	$n > 1.479$, < 1.495
					Quartz (+Silicates)	Tridymite	Cristobalite
A–1	277, 279	Regular	15	1	26	4	70
C–1	102, 156	Regular	14	1	25	4	71
B–1	135	Regular	13	1	40	1	59
A–1	715, 730	Medium	15	1	24	6	70
A–1	1,150, 1,151	Fine	15	1	26	4	70
C–1	934, 1,052	Fine	14	1	26	5	69
A–2	275, 288, 290, 293	Regular	15–15	2	19		
A–3	39, 53, 144, 249	Regular	15–15–15	3	16	19	65
C–3	66, 153, 179	Regular	14–14–15	3	14	20	64
B–3	174, 202, 280	Regular	13–13–15	3	18	10	
A–3	427, 428, 439, 501	Medium	15–15–15	3	13	17	70
A–3	1,028, 1,066, 1,057, 1,046	Fine	15–15–15	3	13	17	70
C–4	12	Regular	14–14 15–14	4	12	30	58
C–6*	74	Regular	14–14–15 14–15–14	6	14	44	42
B–6*	326	Regular	13–13–15 14–15–14	6	12	45	43

* Analysis of No. 74 and No. 326 by V. Dolmage.

The figures given as the proportions of quartz and tridymite in the test brick represent in each case results of measurements taken on 150 to 250 grains. As a check on the work, percentages were calculated for each 75 to 80 grains. The first two results usually agreed within 3 to 4 per cent.; if not, an additional 80 to 100 grains were studied. The figures so obtained are believed to be truly comparative as showing the direction and approximately the velocity of the transformations occurring, notwithstanding the constant errors due to the difficulties above-mentioned, and to the personal equation. The latter errors are believed to be small, since results obtained on the same powder by Mr. Victor Dolmage (who kindly assisted with many of the measurements) and by the writer, usually checked within 3 per cent.

Effect of Variations in Grind and Burning Temperature, and of Repeated Burning, upon Constitution of the Brick

The effect of repeated burning on the constitution of brick of regular grind, burned several times, is shown in Fig. 18. The analyses of this brick after repeated burnings are shown in Table 21.

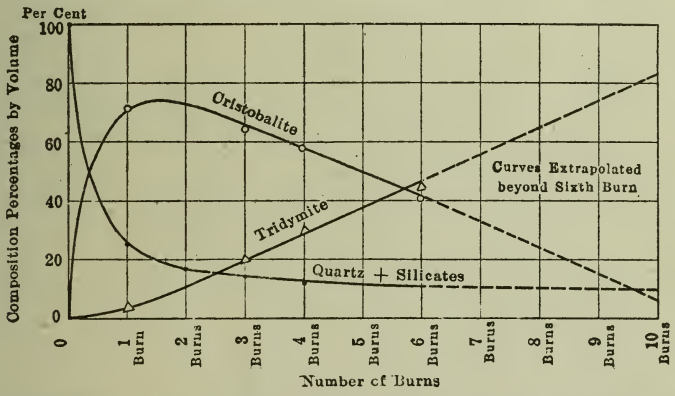


FIG. 18.—EFFECT OF REPEATED BURNING UPON THE CONSTITUTION OF SILICA BRICK. BRICK OF REGULAR GRIND. BURNING TEMPERATURE, CONE 14–14–15–14–15–14.

TABLE 21

Mineral	Volume. Per Cent.					
	Number of Burns					
	1	2	3	4	5	6
Quartz (+Silicates).....	25	....	14	12	....	14
Cristobalite.....	71	....	64	58	....	42
Tridymite.....	4	....	20	30	....	44

It is evident that in the first burn the larger part of the quartz is transformed into cristobalite, and that on repeated burning the remaining quartz is very slowly transformed.[77] The cristobalite at first very slowly and later more rapidly, inverts to tridymite. These results are apparently in accordance with the conclusions of Grum-Grzimailo and Endell, that silica brick heated a sufficient length of time at the proper temperature go slowly but completely over into tridymite.

Time and temperature of burning appear to be the controlling factors. Bricks burned a single time at cones 14 and 15 show essentially the same amount of transformation; those burned at cone 13 show considerably less. The effect of variations in grind is comparatively slight.

Study of Thin Sections

Examination of thin sections cut from certain specimens of test brick show that the inversions proceed most rapidly in the groundmass, most slowly in the larger grains. This is illustrated in Fig. 19, a sketch made from thin section of brick of fine grind, burned three times at cone 15. The groundmass and the rims of the grains are made up of cristobalite and tridymite, with a little residual quartz; the interior of the grains consists of cristobalite and quartz, with a very little tridymite.

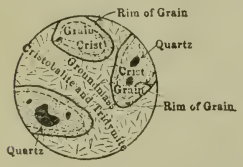


FIG. 19.—SKETCH MADE FROM THIN SECTION OF BRICK OF FINE GRIND, BURNED THREE TIMES AT CONE 15.

Photomicrographs of the thin sections are shown in Figs. 20 to 23 inclusive. The dark areas represent principally cristobalite, the lighter areas, quartz and tridymite. From a comparison of the first two figures with the second two, the increased amount of tridymite in the later burns is at once evident.

THE RELATION OF THE CONSTITUTION OF SILICA BRICK TO THE PHYSICAL PROPERTIES

The Effect of the Addition of Lime

It has already been shown that fineness of grind is not the controlling factor in the formation of tridymite, since that mineral evidently forms nearly as rapidly in the brick of coarse (or regular) grind, as in those of finer grinds. A seeming contradiction exists between this fact and the fact that the inversion to tridymite takes place more rapidly in the fine-grained groundmass of the brick than in the larger quartzite fragments (see Fig. 19).

The relative concentrations of lime in the groundmass of brick of

[77] Compare with results obtained by Seaver, shown in Table 12.

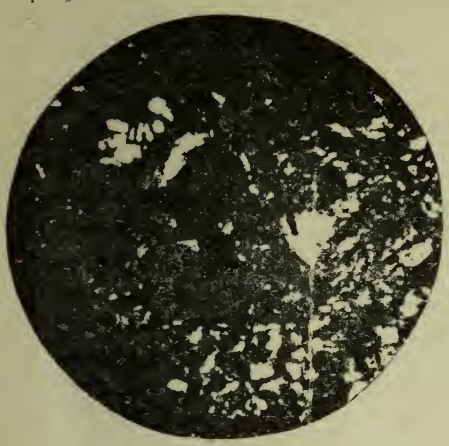


FIG. 20.

FIG. 21.

FIG. 20.—SILICA BRICK OF REGULAR GRIND, BURNED ONCE AT CONE 15 (WHITEHEAD). CROSSED NICOLS. × 120.

The white areas represent residual quartz grains of irregular size and shape, surrounded by a dark field of cristobalite. Little, if any, tridymite is to be seen.

FIG. 21.—SILICA BRICK OF FINE GRIND, BURNED ONCE AT CONE 15 (WHITEHEAD). CROSSED NICOLS. × 120.

The figure shows residual quartz grains surrounded by cristobalite. There are a few narrow, elongated white areas in the field, which may represent tridymite.

FIG. 22.

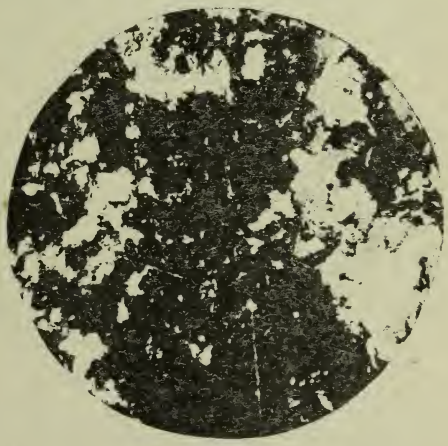


FIG. 23.

FIG. 22.—SILICA BRICK OF REGULAR GRIND, BURNED THREE TIMES AT CONE 15 (WHITEHEAD). CROSSED NICOLS. × 120.

Tridymite and residual quartz are shown by the white areas; cristobalite by the dark areas.

FIG. 23.—SILICA BRICK OF REGULAR GRIND, BURNED THREE TIMES AT CONE 15 (WHITEHEAD). CROSSED NICOLS. × 120.

The larger white areas indicate places in which the conversion to tridymite is nearly complete. The smaller white areas in the darker portion of the field represent residual quartz and tridymite, surrounded by cristobalite (shown in black). Figs. 22 and 23 are taken from different parts of the same thin section.

various grinds probably has some bearing in this connection. It will be recalled that in the manufacture of the brick, 2 per cent. of lime is added to ground quartzite containing about 2 per cent. of impurities (mainly Al_2O_3 and Fe_2O_3). Since all of this enters the groundmass, the proportion of lime therein is much higher than 2 per cent.

The greatest concentration of lime in the groundmass will occur in that brick with the least proportion of fines (*i.e.*, the regular grind) in which, therefore, conditions will be most favorable for the formation of flux compounds during the burning. The catalytic action of the flux compounds probably explains the observed inversion of cristobalite to tridymite, which as Fenner has shown, does not form except in the presence of a flux. According to this point of view, an increase in the amount of lime in the brick should accelerate the formation of tridymite.

Specific Volumes of the Silica Minerals

As an aid in interpreting the results of the microscopic study, use has been made of the specific volume curves of Fig. 24, based upon the following data:

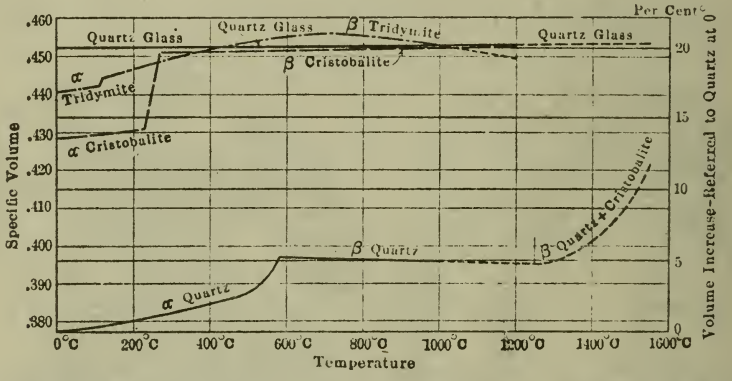


FIG. 24.—SPECIFIC VOLUMES OF THE SILICA MINERALS AND QUARTZ GLASS.

The quartz curve is a reproduction of that shown in Fig. 4, taken from the work of Day, Sosman and Hostetter.

The specific volumes at 20° of quartz glass, tridymite and cristobalite were calculated from their specific gravities, using the values shown below:[78]

	Specific Gravity
Quartz glass	2.210 (Dana, Endell)
Tridymite	2.270 (Fenner)
Cristobalite	2.333 (Fenner)

[78] See Table 3.

The quartz-glass curve was obtained by computing the volume expansion from the linear expansion[79] as given by LeChatelier, and applying these figures to the specific volume at 20°.

The curve for cristobalite is based upon the work of Rieke and Endell, and of LeChatelier. Both show that the expansion of cristobalite from 0° to 230° is slight, and that the volume changes at 230° to 270° is considerable,[80] due to the α to β cristobalite inversion. Rieke and Endell find that the specific volume and coefficient of expansion of cristobalite above 300° are nearly the same as those of quartz glass; the specific volumes, as computed from the expansion data of LeChatelier[79] are considerably lower than those of quartz glass. The view of Rieke and Endell is taken in plotting the cristobalite curve of Fig. 24.

The tridymite curve was calculated from the thermal expansion as given by LeChatelier (Fig. 3). The specific volume of tridymite from 500° to 1,000° is seemingly a little greater than that of quartz glass. While this is probably incorrect, the difference between the specific volumes of the two materials is doubtless small.

The curves of Fig. 24 have been used hereinafter in discussing the probable effect of the changes in constitution upon certain properties of silica brick. It is believed that the inaccuracies of these curves are merely slight errors of degree; and that, while conclusions drawn from them can not be numerically exact, they will serve to indicate the general trend of the changes considered.

The Phenomena of Expansion

Permanent Expansion, Temporary or Thermal Expansion, and the Effect of Rapid Temperature Changes.—The permanent expansion of silica brick after the first burning (in which ±5 per cent. of tridymite and ± 70 per cent. of cristobalite are formed) amounts to 10 to 11 per cent. by volume. The gradual decrease of quartz and increase of tridymite caused by repeated burning, theoretically requires a slight additional expansion after each burn until equilibrium is reached, since the complete transformation of quartz to cristobalite is accompanied by a volume increase of 13.6 per cent.; quartz to tridymite, 16.8 per cent.

At 1,250° C. the specific volumes of cristobalite and tridymite are approximately 14 to 15 per cent. greater than that of quartz. Since the residual quartz of silica brick gradually goes over into the other forms of silica at furnace temperatures, an underburned brick will expand considerably after being placed in use.

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[79] Fig. 3.

[80] Fig. 3; also Fig. 5.

Tridymite expands upon heating much less than does cristobalite. Its specific volume at 20° is 2.8 per cent. greater than that of cristobalite; at 1,000°, nearly the same as that of cristobalite. The gradual transformation of cristobalite into tridymite, shown to occur upon repeated burning of silica brick, probably takes place also in furnace linings. This inversion is accompanied by little, if any, change in volume at furnace temperature.

From the data of Fig. 24, specific volumes at different temperatures were computed for two materials assumed to consist of mixtures of quartz, tridymite, and cristobalite; expansion curves derived from these figures are shown in curves I and II, Fig. 25. Curve I is based upon an assumed composition of 25 per cent. quartz, 70 per cent. cristobalite, 5 per cent. tridymite; curve II on one of 12 per cent. quartz, 43 per cent. cristobalite,

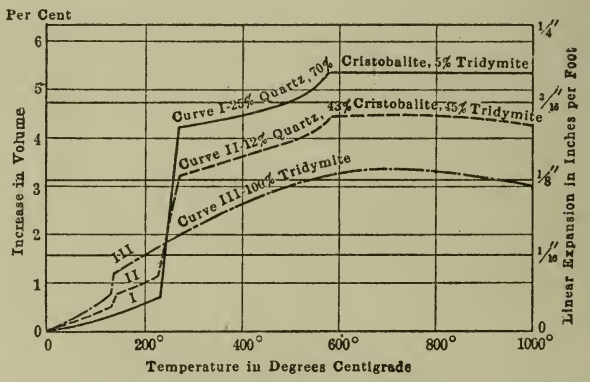

FIG. 25.—THEORETICAL EXPANSION CURVES FOR MIXTURES OF THE SILICA MINERALS.

Composition of Curve I corresponds closely to brick of regular grind *burned once* at cone 14.

Composition of Curve II corresponds closely to brick of regular grind *burned six times* at cone 14–14–15–14–15–14.

42 per cent. tridymite. These figures represent approximately the compositions of brick of regular grind, after one and after six burnings respectively. Curve III is taken from the expansion data of LeChatelier.[81]

Fig. 25 is intended merely to indicate the probable character of the expansion curve of silica brick, and of the effect upon the same of the observed changes in constitution. Obviously, the theoretical curves shown will not correspond precisely with those that would be obtained by actual measurements, being affected by inaccuracies in the data from which they were computed, and, in addition, modified by changes in

[81] Fig. 3.

porosity of the brick, the action of flux compounds, and the lag in expansion due to the temperature gradient between the exterior and interior of the brick.

These curves indicate that the temporary or thermal expansion of silica brick is decreased by repeated burning, and that an all-tridymite brick would have an expansion amounting to about 60 per cent. of that of an ordinary commercial brick, as represented in Curve I.

It is well known that silica brick crack or spall badly if subjected to rapid temperature changes. The phenomenon is probably due chiefly to the rapid expansion at 230° to 270°, accompanying the α to β cristobalite inversion. This effect is diminished by repeated burning. The calculated volume increase at 230° to 270° amounts to 3.5 per cent. for brick burned once, 2.0 per cent. for brick burned six times. An all-tridymite brick should show very little tendency to crack upon rapid heating or cooling, since the only sharp break in the tridymite expansion curve, occurring at 130°, is attended by a volume increase of but 0.3 per cent.

For several reasons, therefore, a large proportion of tridymite in silica brick is desirable, provided the brick are not to be heated continuously above 1,470° C.

1. Tridymite has the least thermal or temporary expansion of the three silica minerals.

2. Tridymite has the greatest specific volume of the three minerals, and can not show any permanent expansion after repeated burning below its melting point.

3. A tridymite brick could probably be subjected to fairly rapid changes of temperature with little danger of cracking.

Comparison of curves I and II seems to show that, while repeated burning produces very desirable effects, the advantages derived up to the sixth burn are entirely disproportional to the additional expense involved, on account of the extreme slowness with which tridymite is formed. Moreover, if used continuously at a temperature exceeding 1,470°, a tridymite brick would slowly revert to cristobalite.

Since the time element appears to be the most important factor in the formation of tridymite, it appears that long-continued burning at the proper temperatures should be more satisfactory than repeated burning. The maximum temperature is maintained 40 hr.[82] in the burning of silica brick; maintaining this temperature for 2½ weeks should presumably give rise to the same changes in constitution as 10 separate burnings under the ordinary conditions of manufacture, and produce brick consisting almost wholly of tridymite (see Fig. 16).

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[82] K. Seaver: *Op. cit.*, p. 136.

Burning Temperature

In order to make a brick containing the maximum proportion of tridymite, the burning temperature should be that at which the inversion to that mineral proceeds most rapidly. It appears that this must occur below 1,470°, since tridymite is not known to form under any conditions above that point. While the inversion proceeds more rapidly at cones 14 and 15 than at cone 13, no conclusions can be drawn from the data herein given as to the most favorable temperature for burning.

Suggestions for Future Study

It seems desirable that the theoretical advantages of a high-tridymite brick should be investigated by comparing the thermal expansion of an ordinary commercial brick with that of a brick high in tridymite. The effect of rapid temperature changes might be compared by means of spalling tests.

If it should be proven that the production of a tridymite brick would be advantageous, the possibility of making such a brick on a commercial basis for certain purposes might well be considered. A small furnace, in which close temperature regulation is possible, should preferably be employed; and the results thereby obtained, if promising, should later be tried out on a larger scale.

The temperature at which the formation of tridymite proceeds most rapidly in silica brick, and the inversion-velocity at that temperature, are as yet undetermined. The possibility of accelerating the inversion by a slight increase in the amount of lime used or by the addition of some other catalyzing material, is worth considering.

It is not impossible that by long-continued heating at the proper temperature, with possibly a slight additional amount of lime, a tridymite brick might be produced on a commercial basis. The results of tests so far made, however, indicate that this is extremely doubtful.

SUMMARY

A brief review is given of the literature pertaining to the silica minerals and the silica refractories.

Microscopic study of the quartzites most important in the American refractories industry shows that they consist essentially of interlocking quartz crystals of random orientation, without a groundmass or cement. This is not in accordance with Wernicke's conclusion that no quartzites are suitable for the manufacture of high-grade refractories except those

which consist of quartz grains in a cement of amorphous silica or cryptocrystalline quartz.

Physical tests and microscopic studies were made of silica brick manufactured under varying conditions of grind, and burned one to four times at slightly varying temperatures. The grinds considered were (1) "regular," or coarse, (2) "shape," or medium, and (3) fine; the burning temperatures ranged from cone 13 to cone 16.

In all cases, a second burning increases the strength of the brick: a third burning has a harmful effect upon the strength in some cases; in others, causes little change. Brick of shape grind are the strongest, those of regular grind the weakest. With a single burning, a temperature of cone 15 produces a stronger material than cone 13 or 14. Uniformity of strength of different specimens of a given lot diminishes upon repeated burning. The most uniform brick are those of regular grind; the least uniform, those of fine grind. Attention is called to the insufficiency of average values as a proper basis for the comparison of strength of non-uniform materials, and for practical purposes the use of certain derived values, termed the "index of uniformity" and the "effective ultimate strength" is suggested.

Upon repeated burning, the quartz, of which the brick is made, is transformed through cristobalite into tridymite. By microscopic methods, estimates were made of the degree of transformation in various bricks. The figures obtained, while not numerically exact, indicate the general trend of the changes considered. The powder studied from brick burned a single time under the ordinary conditions of manufacture consists by volume of approximately 25 per cent. quartz + silicates, 70 per cent. cristobalite and 5 per cent. tridymite; after a sixth burn the composition is approximately 12 per cent. quartz + silicates, 43 per cent. cristobalite, and 45 per cent. tridymite. The inversions proceed more rapidly in the groundmass of the brick than in the larger grains.

It is considered desirable that silica brick should contain a large proportion of tridymite because (1) tridymite has the least thermal expansion of the silica minerals; (2) it will show no permanent expansion after repeated burning; (3) a tridymite brick could probably be subjected to fairly rapid changes of temperature with little danger of injury.

The advantages in these respects theoretically derived up to the sixth burn are believed to be wholly disproportional to the additional expense involved.

SUPPLEMENTARY DATA

Spalling Tests

General Discussion.—It has been shown in the preceding pages that a relation probably exists between the amount of cristobalite in a silica

brick, and its tendency to spall upon rapid change of temperature; and that since the percentage of cristobalite decreases on repeated burning, the spalling tendency should likewise diminish. It was desired to confirm this conclusion through experimental data.

In spalling tests as ordinarily conducted, brick are alternately heated and cooled rapidly a definite number of times between fixed temperature limits; they are usually cooled in the air, but sometimes by plunging into water. The loss of weight in per cent. is taken as a measure of the spalling. This method is not altogether satisfactory, since it does not indicate the internal condition of the brick, which may possibly weaken considerably in the test without much spalling, or lose surface spalls without much weakening; and if the hot test pieces are water-cooled, the formation of steam in the pores may disrupt the brick and lead to inaccurate conclusions.

For the purposes of this study it was decided to make use of a milder heat treatment than that usually employed; one sufficiently severe to cause internal cracks, yet not severe enough to cause spalls to chip off. For each lot of brick studied, the cross-breaking strength was determined upon a certain number of specimens which had been thus treated, and also upon an equal number which had not. The loss in mean strength, expressed in per cent., is considered to represent the "comparative spalling tendency."

Spalling Test in Detail.—The test was carried out as follows: The brick to be subjected to the heat treatment were placed in a gas-fired test kiln, 16 at a time, and heated at a uniform rate of 15° C. per hour (so as to cause no injury to the specimens during the heating) until 600° C. was attained. They were kept at that temperature for 3 hr. and at the end of that time were withdrawn and placed on edge on inverted steel pallets about 2 ft. apart, in a place protected from drafts, and allowed to cool in the air of the laboratory. They were not moved until the following day.

This treatment had the desired effect. No pieces spalled off, nor were the brick visibly shattered; yet when any two brick were struck together they gave a dead ring.

In Table 22 are shown the cross-breaking strengths before and after this heat treatment, of brick of regular grind burned a number of times varying from one to six. The burning temperatures are those shown in Table 14.

In Table 23, these values are summarized and the loss in strength, assumed to represent the comparative spalling tendency, is shown. Figs. 26 and 27 are graphic representations of the data given in Table 23.

TABLE 22.—*Cross-Breaking Tests Made to Determine Spalling Tendency*

Brick Tested on Edge; Supports 6 In. Apart; Load Applied at Midspan

Cross-Breaking Strength of Brick *Not* Subjected to Heat Treatment						Cross-Breaking Strength of Brick Subjected to Heat Treatment					
No. of Burns	Mark	Lot	Cross-breaking, Load in Pounds	Modulus of Rupture		No. of Burns	Mark	Lot	Cross-breaking, Load in Pounds	Modulus of Rupture	
				Modulus, Pounds per Square Inch	Mean					Modulus, Pounds per Square Inch	Mean
1	83	A	4,970	910		1	268	A	1,730	310	
1	279	A	4,820	890		1	295	A	2,480	443	
1	289	A	5,260	960		1	299	A	2,290	412	
1	69	C	4,010	730	848	1	56	C	2,080	373	339
1	71	C	4,790	877		1	73	C	1,900	341	
1	102	C	4,140	750		1	97	C	780	138	
1	156	C	4,520	820		1	166	C	2,000	357	
2	275	A	5,820	1,080		2	18	A	3,430	605	
2	288	A	5,160	980		2	88	A	2,360	421	
2	290	A	3,480	620		2	125	A	3,120	564	
2	293	A	5,140	940		2	240	A	2,030	365	
2	138	B	5,620	1,040		2	196	B	2,440	434	
2	218	B	4,730	860	919	2	22	B	1,600	288	444
2	330	B	6,070	1,120		2	324	B	2,350	420	
2	335	B	3,260	606		2	351	B	2,360	425	
2	10	C	5,420	980		2	96	C	2,310	414	
2	14	C	4,730	880		2	108	C	2,300	419	
2	33	C	5,650	997		2	151	C	2,730	489	
2	110	C	4,950	920		2	318	C	2,740	490	
3	39	A	9,260	1,680		3	70	A	2,850	505	
3	53	A	4,060	740		3	123	A	2,900	518	
3	144	A	5,770	1,040		3	263	A	2,390	425	
3	249	A	7,330	1,340		3	303	A	2,620	468	
3	82	B	6,250	1,100		3	51	B	2,770	495	
3	174	B	7,190	1,310	1,120	3	208	B	3,230	572	489
3	202	B	5,460	990		3	267	B	2,800	494	
3	280	B	3,890	720		3	276	B	3,020	531	
3	66	C	6,290	1,170		3	32	C	3,040	538	
3	78	C	6,480	1,170		3	152	C	1,960	355	
3	153	C	6,380	1,160		3	155	C	2,730	491	
3	179	C	5,470	990		3	171	C	2,650	478	
4	41	A	5,160	947		4	149	A	3,000	547	
4	85	A	6,070	1,100		4	247	A	1,830	325	
4	91	A	3,900	691		4	305	A	2,800	500	
4	238	A	5,600	1,010		4	310	A	1,720	306	
4	188	B	5,170	923		4	89	B	3,040	549	
4	219	B	4,750	848	883	4	140	B	2,150	384	458
4	291	B	4,220	764		4	195	B	2,280	406	
4	329	B	5,480	990		4	350	B	2,050	366	
4	8	C	6,790	1,200		4	22	C	2,950	534	
4	170	C	4,420	789		4	77	C	2,580	471	
4	175	C	3,300	594		4	130	C	2,850	521	
4	313	C	4,150	737		4	148	C	3,230	586	
5	67	A	4,290	769		5	19	A	2,450	434	
5	104	A	4,650	830		5	76	A	2,270	407	
5	147	A	7,360	1,320		5	132	A	3,020	543	
5	158	A	5,730	1,040		5	173	A	2,900	516	
5	186	B	6,270	1,150		5	80	B	3,010	528	
5	215	B	4,920	886	1,050	5	216	B	3,350	601	558
	224	B	6,000	1,060		5	308	B	3,800	691	
5	230	B	5,350	961		5	342	B	2,600	466	
5	257	C	5,950	1,090		5	122	C	2,820	510	
5	262	C	6,920	1,240		5	145	C	3,840	688	
5	306	C	5,950	1,080		5	165	C	3,410	611	
5	338	C	6,650	1,200		5	297	C	3,860	695	
6	4	A	5,620	1,010		6	87	A	3,570	638	
6	201	A	5,230	935		6	126	A	3,170	573	
6	266	A	4,780	850		6	157	A	3,250	572	
6	325	A	5,600	1,010		6	159	A	2,810	504	
6	136	B	5,580	1,010		6	30	B	2,610	468	
6	211	B	5,750	1,030	951	6	185	B	2,640	471	522
6	250	B	5,000	908		6	232	B	1,940	349	
6	255	B	3,680	668		6	320	B	2,750	479	
6	6	C	5,150	916		6	91	C	3,300	600	
6	13	C	3,980	704		6	94	C	2,990	549	
6	64	C	6,590	1,190		6	160	C	2,870	517	
6	150	C	6,560	1,180		6	317	C	3,010	549	

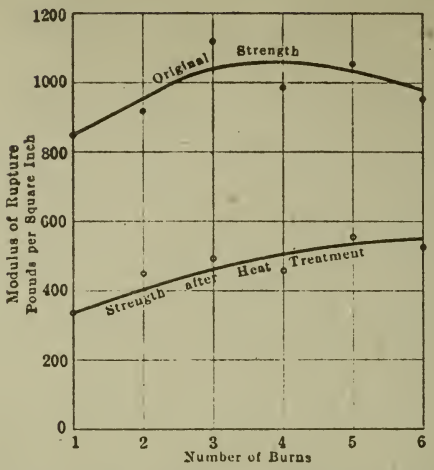

FIG. 26.—VARIATIONS IN THE CROSS-BREAKING STRENGTH OF BRICK OF REGULAR GRIND UPON REPEATED BURNING, AND THE EFFECT UPON THE STRENGTH OF A CERTAIN HEAT TREATMENT, CONSISTING OF SLOW HEATING TO 600° C. FOLLOWED BY COOLING IN THE AIR.

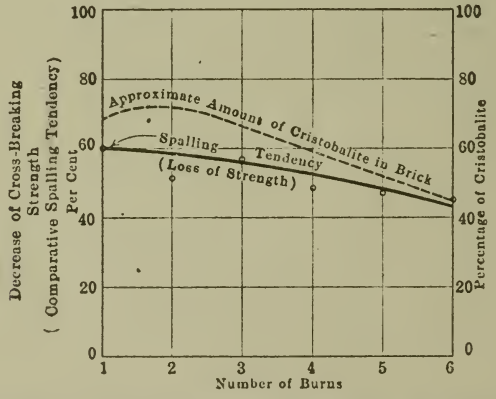

FIG. 27.—DECREASE IN CROSS-BREAKING STRENGTH EXPRESSED IN PER CENT. DUE TO HEAT TREATMENT DESCRIBED. THIS PERCENTAGE DECREASE IS ASSUMED TO REPRESENT THE COMPARATIVE SPALLING TENDENCY.

TABLE 23.—*Summary of Cross-breaking Tests Showing Loss of Strength due to Heat Treatment*

Burn	Modulus of Rupture, Pounds per Square Inch		Loss of Strength due to Heat Treatment	
	Before Heat Treatment	After Heat Treatment	Pounds per Square Inch	Per Cent. (= Comparative Spalling Tendency)
1	848	339	509	60.0
2	919	444	475	51.8
3	1,120	489	631	56.2
4	883	458	425	48.1
5	1,050	558	492	46.9
6	951	552	429	45.1

Results.—The experimental data verify the conclusion that the spalling tendency of silica brick diminishes on repeated burning. In Fig. 27 there is a marked correspondence between the curve representing approximately the cristobalite content of the brick and that representing the spalling tendency. The latter, however, does not decrease as rapidly as the former. It is evident that, while reburning the brick diminishes

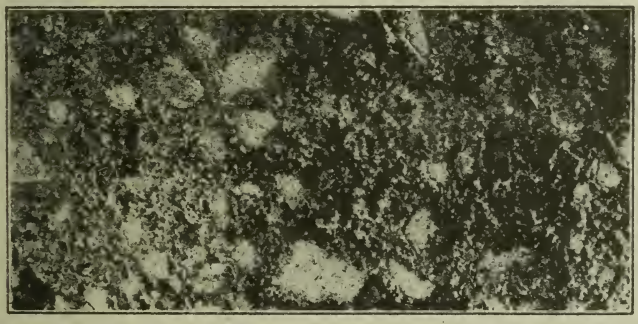

FIG. 28.—PHOTOMICROGRAPH OF POLISHED SECTION OF BRICK OF REGULAR GRIND, BURNED ONCE IN SILICA BRICK KILN. × 4.

the spalling tendency, the change therein from 60 per cent. on a first burn to 45 per cent. after a sixth is too slight to be of any commercial importance.

A number of first-quality fire-clay brick, subjected to the same heat treatment, lost about 4.5 per cent. in strength. This result agrees with the well-known fact that the spalling tendency of clay brick, at the temperature of this test, is but a fraction of that of commercial silica brick.

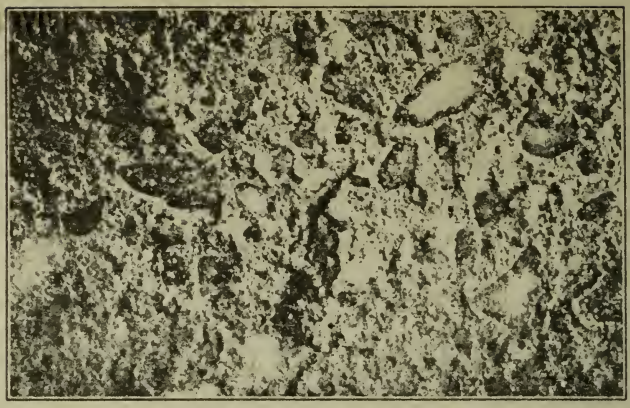

FIG. 29.—PHOTOMICROGRAPH OF POLISHED SECTION OF BRICK OF REGULAR GRIND, BURNED SIX TIMES. × 4.

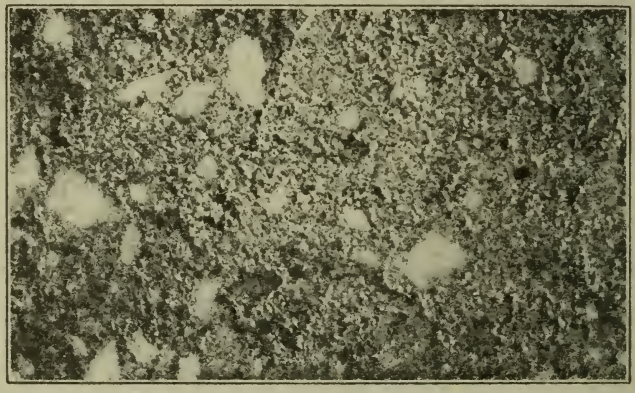

FIG. 30.—PHOTOMICROGRAPH OF POLISHED SECTION OF BRICK OF SHAPE GRIND, BURNED ONCE. × 4.

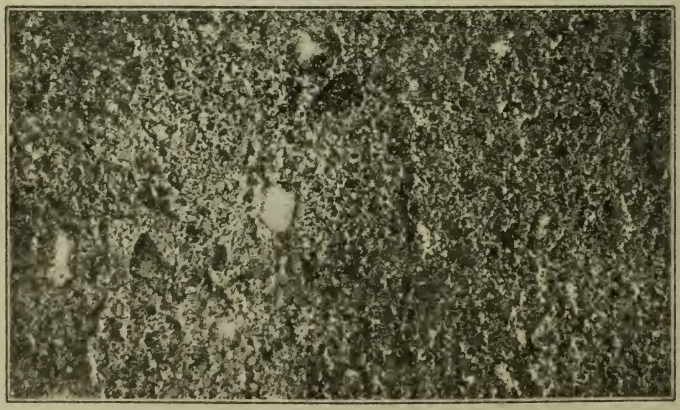

FIG. 31.—PHOTOMICROGRAPH OF POLISHED SECTION OF BRICK OF SHAPE GRIND, BURNED SIX TIMES. × 4.

Changes in Texture of Brick Due to Burning

The photomicrographs shown in Figs. 28 to 31 bring out very clearly the change in the texture of the brick brought about by repeated burning. After a single burning the larger grains of quartzite appear nearly white; after six burnings the grains stand out much more distinctly in the pictures and have become darker and apparently more dense. A number of grains are seen in which the center is still white, while the margins are altered in the manner described.

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